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# Japan Report

## SCIENCE AND TECHNOLOGY

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# JAPAN REPORT

## SCIENCE AND TECHNOLOGY

## CONTENTS

AEROSPACE SCIENCES

# DEVELOPMENTS IN DEFENSE, AIRCRAFT INDUSTRY, SPACE UPDATED

November 1986

Tokyo AEROSPACE JAPAN in Japanese Nov 86 pp 28-30

[Excerpts] DEFENSE

## Eleven Aircraft Projects Funded

The TRDI (Technical Research and Development Institute) of the Defense Agency has renewed funding for 11 aircraft-related research and development articles totalling Y10 billion in the budget for fiscal 1987. These articles are detailed below:

(Development Article)

1. ECM (electronic countermeasures) equipment ALQ-7
2. Tail warning equipment

(Research article)

1. Sea mine scattering equipment for transports
2. Helicopter rotor hub
3. Trial-manufacturing of RCS reducing model
4. Trial-manufacturing of RPV (remotely piloted vehicle)
5. Trial-manufacturing of reheating fan engine
6. Flight maneuvering environment simulator
7. Trial-manufacturing of joint electronic warfare system
8. Tow target
9. Performance rising type sonobuoy

## Performance Rising Type F-4 Explained at Security Meeting

The Defense Agency explained a plan for modifying the F-4EJ at a security meeting held on 12 September. The security council will deliberate on and approve this plan.

An F-4EJ was modified on trial with a view to increasing its performance during the period from fiscal 1983 to 1984. The APW (Air Proving Wing) of the

ASDF (Air Self-Defense Force) conducted tests on this modified F-4EJ, and confirmed the increase in performance during the period from the latter half of fiscal 1984 to the first half of fiscal 1985. The Defense Agency has proposed to the Ministry of Finance a budget whereby the modification at first of 10 F-4EJs will be started on a full scale on the basis of the above result at a cost of Y17.8 billion in fiscal 1987. The Defense Agency explained the necessity and plan for modifying these F-4EJs to the security council, because this equipment is important to the modification of the F-4EJs is a work of interceptor fighters.

The contents of the explanation have not been disclosed, but a projected outline is given below.

1. It is expected that the fire control capacities such as look-down capacity, control calculation capacity, and display capacity will be increased by mounting the APG-66 as a new radar equipment instead of the APG-120, a domestically developed central computer, and a new HUD (head-up display) Kaiser 36500-01) on the F-4EJ. It is expected that the missile mounting capacity will be increased by again mounting the AIM-7F/9L and ASM (air-to-surface missile)-1 on the F-4EJ. It is expected that the navigation capacity will be increased by mounting the Litton LN-39 as a new INS (inertial navigation system) instead of an old one on the F-4EJ. It is expected that the IFF (identification friend or foe) capacity will be increased by newly mounting an IFF APX-76 on the F-4EJ. It is expected that the radar warning capacity (expansion of frequency bandwidth) will be increased by mounting the APR-4 Revision as new radar warning equipment instead of an old one on the F-4EJ. As a result, the intercepting capacity of the F-4EJ will still be inferior to that of the F-15, but will be greatly superior to that of the old F-4FJ. In addition, the F-4EJ will have the support fighting capacity which surpasses that of the F-15, because it will be armed with ASM-1s.

2. All the present F-4EJs will not be modified, but 96 F-4EJ's will be modified except for 2 F-4EJs for schools and 17 F-4EJs which will be modified as reconnaissance aircraft. Therefore, it will be possible to organize four flight squadrons each consisting of 22 F-4EJs. Ten F-4EJs will be modified at the first fiscal year, and subsequently, about 20 F-4EJ's will be modified every year for a period of 7 years extending to fiscal 1993.

Completion of C-1 Equipped with FCS

On 26 September KHI (Kawasaki Heavy Industries, Ltd.) delivered in the TRDI the C-1, a flight experimental aircraft equipped with a future FCS (fire control system), and the TRDI requested the APW of the ASDF to cooperatively conduct performance checking tests on the C-1.

Research on this future FCS has been conducted since around fiscal 1980 with the aim of mounting the system on future fighters. Equipment materials for the system were completed by MEC (Mitsubishi Electric Corp.) in this fiscal year, were immediately delivered in KHI, and were mounted on the C-1. The modification work accompanied with the mounting of these equipment materials and the FCS on the C-1, covers only the internal sections of the C-1.

Therefore, in appearance, the C-1 is almost the same as conventional C-1s, because an antenna is also incorporated in the radome.

The APW of the ASDF will start conducting flight tests on the C-1 in October, and is scheduled to conduct about 110 flights by the end of fiscal 1987. This FCS has technically come into the limelight, because it employs an active phased array radar equipment for the first time in the world.

AIRCRAFT INDUSTRY

NLA Investigates Vitality of Helicopters

On 26 August the NLA (National Land Agency) requested the JAA (Japan Aeronautic Association) to investigate vital policies for aircraft such as helicopter, etc., with a view to promoting a stay concept, and the JAA decided to establish a committee consisting of knowledgeable and experienced consultants and members of relevant ministries and agencies. The committee expects to meet in December, and will make an interim report on the investigation. The investigation results will be summarized in March of the next year. Also, working groups under the leadership of the chairman of the committee will be established. It was decided that the first working group would study the use of aircraft such as helicopters, etc., the second working group would study the establishment of heliport, etc., and the third working group would study the measures for promoting local areas by conducting case studies, repsectively.

Also, Azeo Sakakibara, a professor of the Department of Economics at Doshisha University has been inaugurated as chairman.

Helicopter Business, 100,000 Hours Per Year

The change of operating time of Japanese helicopter business companies during the period 1981 to 1985 is as follows: 99,625 hours in 1981, 96,816 hours in 1982, 99,697 hours in 1983, 102,153 hours in 1984, and 104,030 hours in 1985, respectively. That is, it has almost been leveling off at an operating time of about 100,000 hours since 1981. The business content is as follows: 1) the time for both vertical and oblique photography has been increased since 1981; 2) the time for gathering news is leveling off; 3) the time for scattering chemicals was decreased for the first 3 years from 1981 to 1983, but it has been increased since 1984, and it reached a maximum of 44,000 hours in 1985; 4) the inspecting and investigating time and the controlling and training time are almost leveling off; 5) the work of transporting passengers between two areas was begun in 1985 for the first time, and the time for the work is 1,711 hours; 6) the time for sightseeing flights was increased; 7) the time for construction cooperation was decreased annually, with 1985 time being only 65.9 percent that of 1981.

Helicopter Commuter Investigation Committee Starts

On 25 September the SJAC (Society of Japanese Aerospace Companies, Inc.) held the first meeting of the Helicopter Commuter Business Investigation Committee at the KKR Tokyo Takebashi.

The committee established a commuter aircraft industrial study subcommittee last year. As a result, it came to the conclusion that the work of transporting commuters by fixed wing aircraft in Japan cannot avoid confronting a problem of constructing airports and the problem of the limits of extending of helicopter service into existing airports. On the other hand, there is room for further studying the extension of the helicopter service, because unlike fixed wing aircraft, helicopters do not need a large airfield. The committee decided to narrow the argument about transportation of commuters by helicopters with consideration to the above matters, and decided to study a commercializing plan on the assumption of some model projects which are promising for helicopter service. In addition, the committee regarded Iwate, Ishikawa, and Kyoto as model areas, and decided to make a report in about a year, around October 1987. Also, Akira Azuma, a professor of the University of Tokyo has been inaugurated as chairman of the committee.

Aircraft Industry in 1986

MITI (Ministry of International Trade and Industry) has arranged data on present status of Japanese industry in 1986; the data include the aircraft industry as shown below.

1. The aircraft industry is formed by collecting high-technologies from various fields such as electronic technology, mechanical technology, etc., and can be called, "Joint Technical Industry." It is also a knowledge intensive high-level assembly industry. In addition, it is an industry which will develop increasingly even in countries poor in resources, like Japan, because it has a high value added, and the amount of raw materials used per person is small. The aircraft industry has grown on a large scale in advanced countries in the West. But, the sales of the Japanese aviation and space industry are only about 2 percent of those in the entire world, and are one-twentieth those in the United States, one-third those in France and England, and two-fifths those in West Germany.

2. The production amount (manufacturing and repairing) of the aircraft industry in 1985 is Y620.2 billion, which is 22.4 percent above the comparable figure for the previous year, and has reached the scale of Y600 billion mark for the first time since the end of the Pacific War. Most of the produced aircraft are delivered in the Defense Agency, and account for 84.6 percent of all the produced aircraft. Also, the work of repairing these aircraft accounts for 73.3 percent of the entire aircraft repairing work. The amount of aircraft exported in 1985 was Y46.7 billion (4.2 percent above the comparable figure for the previous year). The amount of aircraft produced for Japan in that year was Y573.5 billion (12.0 percent above the comparable figure for the previous year). The amount of aircraft imported in Japan in that year was Y359.3 billion.

3. In order to develop the aircraft industry, it is necessary to continuously make every effort to develop the airframe, engine, etc. In the future, the direction of the technical development will turn to the energy saving and low-environmental pollution airframe in a short term. Therefore, it has become important to widely improve engines and to develop peripheral systems. The aircraft technology has a large influence on various fields, but the aircraft

development work is accompanied with technical difficulties, and needs the tremendous funds and long period of time. For this reason, foreign countries properly take the horizontal division of labors such as international joint development work, etc., for the purpose of ensuring the market for aircraft and diversifying the risks and aircraft work.

MHI Receives Order for Research on Reduction in RCS

MHI (Mitsubishi Heavy Industries, Ltd.) has received from the Defense Agency an order for research and trial-manufacturing of a mock-up for reducing the RCS, priced at Y65,739,000. The delivery date is 31 March 1987.

This is a research enabling the shape of aircraft which cannot be readily found with radar equipment is considered by reducing the RCS, and is the first research for the TRDI. The past data indicate that radar reflecting characteristics are satisfactory, and in other words, the sections which can be readily shown up on the radar screen are the leading edge of main wings, air-intakes of the fuselage, etc. In fiscal 1986, MHI will conduct research on main wings, and intends to make one of the main wings of a fighter on an experimental basis and to conduct tests on this main wing while applying radar waves to the main wing at a testing ground. In addition, in fiscal 1987 the company will make a model of the air-intakes, will conduct research on the model, and will aim at reducing the RCS to 10 decibels for the time being and to 30 decibels finally. Ten decibels means that compared with a conventional case in which no measure is taken, radar equipment will not work unless the detecting distance of the radar equipment is close to about 60 percent. Thirty decibels means that such radar equipment will not work unless the detecting distance is close to almost visibility.

SPACE DEVELOPMENT

New Liquid Hydrogen Plant Established

Showa Denko K.K. has established a joint concern in cooperation with Rail Liqude Group in France, which supplies liquid hydrogen as a fuel to the Ariane Rocket of the ESA (European Space Agency), and has constructed a plant with an annual production capacity of 7,400 kiloliters in the Oita Complex of Showa Denko K. K. The plant will start producing liquid hydrogen in June 1987. Showa Denko K. K. estimates that the sales for the first fiscal year will be about Y1 billion.

Also, Japan Liquid Hydrogen Co., Ltd. which is a joint concern set up by Iwatani & Co., Ltd. and MHI, is constructing a new plant with an annual production capacity of 10,000 kiloliters in the vicinity of TNSC (Tanegashima Space Center) of NASDA (National Space Development Agency of Japan) in Kagoshima Prefecture. The new plant will start producing liquid hydrogen in December 1987. Up to now, Tashiro Field Laboratory of MHI in Akita Prefecture has produced liquid hydrogen with a view to performing experiments on fuel. But, the production capacity of the new plant will be more than twice that of Tashiro Field Laboratory.

December 1986

Tokyo AEROSPACE JAPAN in Japanese Dec 86 pp 26-28

[Excerpts] AIRCRAFT INDUSTRY

U-36A1 Will Conduct Test Flight

Shin Meiwa Industry Co., Ltd. conducted the first flight of the U-36A1, a trainer support whose order was requested from the MSDF (Maritime Self-Defense Force) to the company, on 18 October. The trainer support took off from the Tokushima Airfield, and Mr Baba, a pilot controlled it. Following the first flight, the company conducted the primary performance checking flight for three cycles over a period of 2 weeks, and completed the flight test program for the year. It was decided that the secondary flight test would be conducted at the beginning of the next January.

In March 1985 the company received from the Defense Agency an order for remodeling a prototype Gates Learjet 36 into the U-36A1. The company received such an imported Gates Learjet 36 from the Defense Agency in December 1985, and has remodeled it into the U-36A1. Through November to December the company removed electronic equipment from the aircraft, installed this electronic equipment in a mock-up, conducted overall operating preliminary tests on the mock-up, removed the electronic equipment from the mock-up, installed them and training equipment in the aircraft, and conducted ground functional tests on the aircraft. This training equipment were not installed in the aircraft at the primary flight tests. The company is scheduled to start conducting the test flight of the fully equipped aircraft at the beginning of January 1987, and after conducting the in-house flight tests on the aircraft until March, the company is scheduled to deliver it to the Defense Agency.

Paris Show in 1987: the Number of Japanese Companies Is Doubled

The SJAC has decided to participate in the 1987 Paris Air Show which will be held at the Le Bourget Airport in a suburb of Paris in June 1987. This is the 10th time that the SJAC has participated in the Paris Air Show.

The SJAC has applied to participate in the show in expectation that the number of indoor exhibits and booths will be about twice that in the previous show. The area necessary for these indoor exhibits has been increased from 194 square meters to 365 square meters, and that necessary for these booths has doubled from the previous 72 square meters to 144 square meters. The list of companies exhibiting has not yet been closed, but it is certain that the number will surpass the 19 at the previous show.

Japan Also Studies Joint Development of MPC75

Arao, representative director and Tateyama, managing director of the SJAC visited China from 3 to 8 October, and exchanged views with members of the CATIC (China Aerotechnology Import and Export Corp.). A problem of joint development and production of 30 or 40 seat commuter airplanes has been discussed with Chinese members since last year. But, this problem has been shelved for the present, because it has been clarified that the number of airplanes does not reach the demand number aimed at the Sino-Japanese mutual market; the time is not ripe for promoting joint development and production plan. The CATIC tries to develop the MPC75, a 60 or 85 seat passenger airplane in collaboration with MBB (Messerschmitt-Bolkow-Blohm) in West Germany. The CATIC has recommended Japan participate in the development plan instead of the above plan.

For this reason, the SJAC will hold a China Project Promotion Special Committee (chairman: Yoshio Sasaki, chief director of Aircraft and Special Vehicle Headquarters of MHI) and will study this new plan in the near future.

New Japan Aircraft Maintenance Receives Order for Lavatories of 747-400

New Japar Aircraft Maintenance Co., Ltd. has received from Boeing Corp. an order for developing and producing lavatory modules which will be installed in the 747-400.

Details of this installation work have not been clarified, but it has been decided that 14 or 15 sets of lavatory modules will be installed in each 747-400. New Japan Aircraft Maintenance Co., Ltd. has received these sets of lavatories modules for 250 747-400's. The company is scheduled to start delivering such products in Boeing Corp. in October 1987, and it is said that the amount of the order received is Y17.5 to 18 billion.

ANA Aircraft Maintenance Plant Approved by Aerospatiale

The plant of ANA Aircraft Maintenance Co., Ltd., has been approved as a category B plant by Aerospatiale Societe Nationale Industrielle in France. On 27 October the certificate was handed over from G. Raschiero, product support vice-managing director of the Helicopter Division of Aerospatiale Societe Nationale Industrielle to Tsutsumi, president of ANA Aircraft Maintenance Co., Ltd.

Plants approved as Category B plants can repair important sections which require advanced technologies as well as sections specified in usual overhaul manuals. Only eight companies including ANA Aicraft Maintenance Co., Ltd. in the world have such plants.

MHI Develops Data Bus

MHI has succeeded in developing an optical data bus system which will be mounted on aircraft, in cooperation with NEC Corp., and announced that they had established a technology for putting the system into practical use.

Compared with the electric signal transmission system used in aircraft, this optical data bus system is resistant to lightning and thunder, has a little influence on electronic equipment, and can further enhance flight safety. MHI has established the technology for putting the new system into practical use by conducting flight tests on the MHI's own MU-300 equipped with it. This is the first time that such flight tests have been conducted in Japan. Also, the company has conducted different flight tests on the aircraft equipped additionally with a hybrid navigation system consisting of the combination of GPS (global position system) and INS (inertial navigation system).

DEFENSE

F-15 Deployed in Komatsu Air Base

On 1 November the ASDF (Air Self-Defense Force) announced that two F-15s, interceptor fighters would be deployed in the 303d Tactical Fighter Squadron of the 6th Air Wing in the Komatsu Air Base. The 6th Air Wing has the 303d and 306th Tactical Fighter Squadrons, and both squadrons had been armed with only the F-4EJ. First the 303d Tactical Fighter Squadron will be armed with the F-15 instead of the F-4EJ, and in about 1 year all aircraft deployed in this squadron will be changed from the F-4EJ to the F-15.

Up to now, F-15s have been deployed in two fighter squadrons (the 201st and 203d Tactical Fighter Squadrons) in the Chitose Air Base, one fighter squadron (the 204th Tactical Fighter Squadron) in the Hyakuri Air Base, and one fighter squadron (the 202d Tactical Fighter Squadron) in the Nyutabaru Air Base, respectively. In the past, all the squadrons had been armed mainly with the F-104J. But at present, there is no longer any fighter squadron armed with the F-104J. Following on from the above change from the F-104J to the F-15, the F-15 will replace the F-4EJ deployed in the 303d Tactical Fighter Squadron. When this squadron is armed with the F-15 instead of the F-4EJ, it will become the fifth squadron armed with the F-15. After the 306th Tactical Fighter Squadron is armed with the F-15 instead of the F-4EJ in 1989, a new squadron called, "the 7th Tactical Fighter Squadron" armed with the F-15 instead of the F-4EJ will probably be organized in about 1993.

Proposal on FS-X From Two Companies

From 13 to 17 October, members of the ASO (Air Staff Office) listened to a proposal on FS (fighter support)-X from Alexander Marshall, vice-president in charge of the International Marketing Department of McDonnell Douglas Corp., and other members of the company; a question and answer session followed. In addition, from 20 to 24 October, the above members of the ASO listened to a proposal from D. J. Wheaton, vice-president in charge of aircraft marketing of the Fort Worth Division of General Dynamics Corp., and other members of the company; a question and answer session followed.

The contents of these discussions have not been clarified, but after the members of the ASO finished listening to the two proposals, they summarized and studied the contents, comparing them with those of a domestic development proposal on FS-X, offered by the TRDI.

Development of After-Burner for F-3

The TRDI has appropriated the cost of research and trial manufacturing of a re-burning fan engine in the budget for fiscal 1987. This re-burning fan engine is so called, "afterburner" for jet engines. It has been decided that the TRDI will conduct tests on a subsonic F-3 engine equipped with a supersonic afterburner experimental unit. Originally, this subsonic F-3 engine was developed for the T-4. The thrust is scheduled to increase to 1.5 times that of present engines.

Articles which will be made on an experimental basis are the combustion chamber, duct, exhaust nozzle, fuel control, etc. Articles which have been put to practical use, are equipped with nozzles having an automatic opening and closing system. The above articles are equipped with nozzles having no such system, because they are test articles. Assuming that the TRDI starts carrying out the above tests and trial manufacturing work in fiscal 1987, the TRDI is scheduled to finish them in outline in fiscal 1990.

Also, when the J79 engine equipped with an afterburner is used in a F-4 Phantom jet fighter, the ratio of the thrust to that of an original engine will be from 1.3 to 1.4:1. When such an engine is used in the F-15 Eagle jet fighter, it will be 1.5:1. The TRDI will make efforts to obtain the 1.5 factor.

Service Tests on AQM-1 Are Finished

Fuji Heavy Industries, Ltd. is developing the target drone, XJ/AQM-1 for the ASDF. Two target drones were delivered in the ASDF in November, and the number of target drones which will be subjected to service tests is five. It has been decided that these target drones will be subjected to air launching tests by December.

Of the five target drones which will be subjected to service tests, the No 2 target drone was subjected to the first program flight test and was attacked with the Sparrow, an AAM (air-to-air missile), on 11 November. These service tests will be performed by using the Sparrow and Sidewinder AAMs until December.

SPACE DEVELOPMENT

Two Committees Are Active in SJAC

On 13 October the SJAC held the First Basic Technology Investigation Committee and the First Legal System Study Committee concerning space use with a view to promoting such use.

It has been decided that both committees will study various items as working groups of the Space Industry Use Special Council of MITI. Respective committees will be held at a rate of about once a month, and members of these committees are scheduled to prepare reports in March 1987.

Also, the Basic Technology Investigation Committee for promoting space use takes action as a technical study working group of the Space Industry Use Special Council, and consists of 20 members and the chairman, Isao Kudoh, chief of Space Environment Technical Research Room of Limit Technical Department of Electrotechnical Laboratory of MITI's Agency of Industrial Science and Technology. The Legal System Investigation Committee concerning space use takes actions as a legal system study working group of the Space Industry Use Special Council, and consists of 11 members and the chairman, Kitaroh Wada, professor of the Department of Domestic Science of Showa Women's University.

FEO Requires Increase in Budget for Space

Morikawa, managing director of the Development Department of the FEO (Federation of Economic Organizations) mentioned that the FEO Space Development Promotion Council had studied an ideal Japanese space budget for the 5 year period of fiscal 1987 to 1991, at the Space Environment Use International Symposium held on 16 October. As a result of the study, this council estimated that Y300 to 400 billion would be required annually and Y1.9 trillion would be required as a budget for the Japanese space industry during the 5 years. According to this estimation, of the high-technology fields, the space development field needs the very long period of term, huge amount of money, and risky investment of funds. Originally, it is difficult to establish the industrial foundation and to promote the technical development work unless the space development field greatly depends on the national budget. Immediately, the government should work out a mid-term execution plan concerning the space development work on the basis of the financial background of the entire country, and should aim at appropriating Y300 or 400 billion per year in the national budget in this mid-term execution plan.

Japan-U.S. Convention of Space Station

With regard to the operation and development of the space station plan and the use stage (phase C/D/E) of the space station, the Third Japan-U.S. Governmental Convention Symposium was held at the Ministry of Foreign Affairs in Tokyo on 13 and 14 October. In the same way, with regard to the space station plan, the Third Japan-U.S. Governmental Convention Execution Organization Symposium was held in Kyoto on 15 and 16 October.

Following the Second Japan-U.S. Governmental Corvention Symposium and the Second Japan-U.S. Governmental Convention Execution Organization Symposium held in Washington, D.C. in September 1986, the above symposiums were held for the purpose of discussing the Japan-U.S. cooperation concerning the development, operation, and use stage (phase C/D/E) of the space station which the United States wants to set about in May 1987. The purposes are to construct a lasting and multipurpose space station in the circumterrestrial orbit at a low altitude of about 500 kilometers by the mid-1990's and to conduct various experiments and observations.

Also, the attendees of the above symposiums were members of the STA (Science and Technology Agency) and the Ministry of Foreign Affairs on behalf of Japan

and the Department of State and the NASA (National Aeronautics and Space Administration) on behalf of the United States, respectively.

Chinese Space Industry Is Investigated

The SJAC dispatched a delegation from the space industry to China in order to have the delegation investigate the contents of the present status of Chinese space equipment, satellite launching work, etc., from 22 to 31 October.

The delegation consists of 13 members including Hiroshi Morikawa, leader of the delegation and managing director of the Electronic System Business Department of Mitsubishi Electric Corp., and visited the Ministry of Astronautics Industry, China Great Wall Industry Corp., Institute of Space Technology, Pneumatic Research Institute, Satellite Assembly Plant, Engineering Environment Test Center, and Rocket Engine Test Center in Beijing, the Shaanxi Province Meteorological Bureau, Wireless Technology Research Institute, and Fine Electron Research Institute in Xi'an, and the Shanghai Aeronautic Bureau, Broadcasting Equipment Plant, Satellite Engineering Research Institute, Xinyu Cell Factory, etc., in Shanghai. The delegation carried out the following investigations and studies by inspection in accordance its schedule, and has already returned to Japan.

1. Investigation of system, scale, etc., of Chinese space development;

2. Investigation of definite contents of satellite launching work;

3. Study by inspection in satellite manufacturing assembly factories and environmental test facilities;

4. Study by inspection in rocket manufacturing assembly factories and combustion test facilities;
5. Investigation and study by inspection in other relevant items and relevant facilities.

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# STATUS, PROSPECTS FOR PROTEIN ENGINEERING

Tokyo BIO INDUSTRY in Japanese Aug 86 pp 46-53

[Article by Daisuke Khoda, researcher at Physiological Active Agent Department, Tokyo General Research Institute of Clinical Medicine, and Shigeyuki Yokoyama, assistant at Biochemical Course, Department of Science, University of Tokyo; first paragraph is editorial introduction]

[Text] What has to be done now is the systematic implementation of experiments that could provide core principles of protein engineering. In this article, results of the latest research on aminoacyl tRNA synthetase will be outlined, with substrate recognition and the role of hydrogen bonds in enzyme reactions being examined in quantitative terms. Lastly, a new method of NMR will be discussed, which will be useful in future protein engineering.

## 1. Introduction

### Protein Engineering Pessimism?

Protein engineering technology refers to design proteins with desired functions, characteristics, and structures, mass produce at low cost. The word protein engineering has been heard more often since genetic DNA was artificially synthesized, and technology to substitute bases in specific parts of genes was developed using the short synthesized DNA. Reflecting the fact that DNA can be handled freely now, there is a tendency to think that the next target will be proteins. The production of order made proteins, which used to be nearly impossible, has suddenly taken on a touch of reality.

However, it should be pointed out that DNA and proteins involve technologies of much different levels. As far as DNA is concerned, the same technologies to cut DNA with restriction enzymes, to determine base sequences, and to close DNA, can be applied to all genetic DNA in the same way, no matter from what species it is taken. Today's genetic engineering includes useful technology which can be applied widely. On the other hand, research on protein engineering so far has not gone beyond describing each protein, because the characteristics of proteins vary a lot. Therefore, knowledge on generally applicable principles is totally lacking. What is worse, technology is being developed through trial and error, and there are very few methods that guarantee success.

In this context, it is unlikely that protein engineering will soon experience as glorious a development as genetic engineering which enabled mass production of peptidergic hormones. Some people have "protein engineering pessimism."

Accumulation of Basic Data Is Necessary for Protein Engineering

There is no doubt that protein engineering will succeed in the near future. What has to be done is systematic implementation of experiments that could provide core principles for protein engineering.

The research to be discussed in this article deals mainly with aminoacyl tRNA synthetase as an object of experiments. A brief explanation of aminoacyl tRNA synthetase is given below.

2. Aminoacyl tRNA Synthetase Is Used for the Most Advanced Protein Engineering

Aminoacyl tRNA synthetase (ARS) is a generic name for a group of enzymes that bond amino acids to tRNA and serve as a catalyst for aminoacyl tRNA synthesis. In principle, there are 20 kinds of ARSs corresponding to 20 kinds of amino acids. These enzymes aminoacylate tRNA through the following two reaction phases:

$$E + AA + ATP \rightarrow E \cdot AA - AMP + PPi$$

$$E \cdot AA - AMP + tRNA \rightarrow AA - tRNA + AMP + E$$

Amino acids (AA) react to ATP and are activated as aminoacyl AMP (AA-AMP) bonds to an enzyme (E). Then, the aminoacyl radical is transferred to tRNA correspondent of the amino acid, and aminoacyl tRNA is used as the supply source of amino acids for protein synthesizing reactions by ribosomes.

The anticodon parts of tRNA are complementary with mRNA codon. Codon is translated into amino acids by the two phases of aminoacylation of tRNA by ARS, and the mutual reaction of codon--anticodon. The 20 ARSs guarantee accurate translation of genetic information by recognizing their specific amino acid and tRNA very strictly.

ARSs are the most sophisticated enzymes in the area of protein studies in that they serve as the optimum experimental strain to observe mutual reactions between low molecular substrates and proteins, and between macromolecular nuclear acids and proteins.

Tyrosyl tRNA Synthetase

A British group led by Winter and Fersht cloned tyrosyl tRNA synthetase (TyrRS, 2 x 47.5 kDa) from the moderate thermophilic bacterium, Bacillus stearothermophilus, and manifested it in colon bacilli, and thus obtained a large amount of the enzyme. Thermophilic bacteria refer to bacteria living in a high temperature environment, and moderate means that they like a high temperature to a moderate extent (the highest temperature for growth

is 55-75°C). Origins of proteins of thermophilic bacteria are known to be stable against organic solvents, denaturation agents, as well as heat. This renders enzymes of thermophilic bacteria very suitable in terms of structure for research on protein engineering.

In order to utilize protein engineering most effectively, the three-dimensional structure of proteins to be reformed should be known. It is desirable that the structure of the complex with a substrate be elucidated and the mutual reaction between the protein and the substrate be known, if possible.

The three-dimensional structure of TyrRS was elucidated by Blow's group from Great Britain by X-ray crystal analysis at 3.0 Å resolving power (Figure 1). Furthermore, the three-dimensional structure of the tyrosyladenylic acid (Tyr-AMP) and TyrRS complex was defined by 2.7 Å resolving power, and it is inferred that there are 11 hydrogen bonds between the substrate and the enzyme (Figure 2). The side chain of amino acid residue of the enzyme is concerned with nine of the bonds.

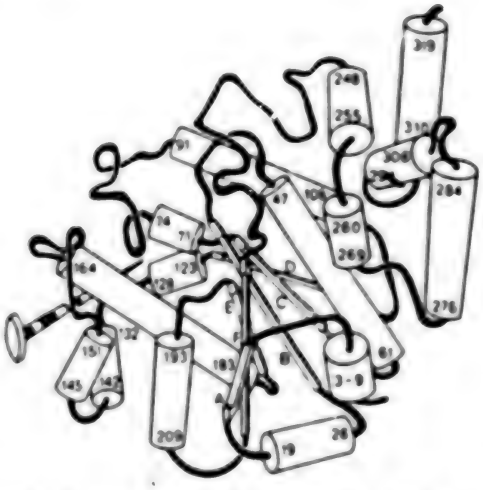

Figure 1. Three-Dimensional Structure of Tyrosyl tRNA Synthetase of Bacillus stearothermophilus, a Moderate Thermophilic Bacterium

1-320 residues from the N-end of the subunits are shown.
99 residues on C side could not be interpreted due to disorder in the crystal.
A two-fold symmetry axis is also shown.

If, like above, a protein or its mutual reaction to a substrate is defined by high resolving power, display by computer graphics is highly effective. It is not only a means of display but also an indispensable method that enables simulation of the effect of amino acid substitution and the mutual reaction between a substrate analogue and a protein, and thus aides protein designing. (Even if the three-dimensional structure can be imagined by using computer graphics, provided that the three-dimensional structure of another protein with similar amino acid sequence is known. Reference 6 concerns this.)

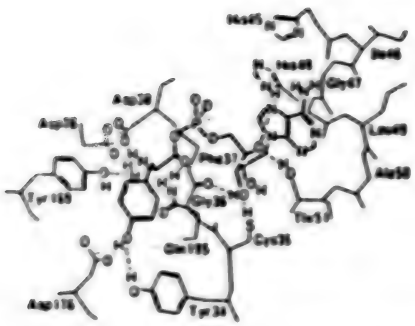

Figure 2(a) Structure of Tyrosyl tRNA Synthetase Near Its Active Part

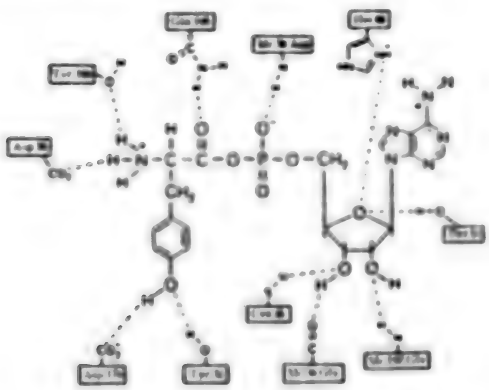

Figure 2(b) Hydrogen Bond Between Tyrosyladenylic Acid and Tyrosil tRNA Synthetase
MC = main chain

TyrRS is the first enzyme that was studied with part specific mutation introduction method by using oligonucleotides. Since TyrRS was reported in 1982, more than 100 transformants have been produced to date, and their enzymological characteristics are being studied.

Three amino acid residues that are considered to have hydrogen bonds where the ribose parts of adenylic acid, namely, Cys 35, His 48, and Thr 51, were substituted, the activity of amino acid activator, a decrease or increase in aminoacylation activity was observed (Table 1). When Tyr 34, which has hydrogen bonds with OH radicals of substrate tyrosine and is considered to play an important role in substrate specificity, was substituted with Phe 34, the specificity of amino acid activation of substrate tyrosine to substrate phenylalanine was slightly lowered.

3. Role of Hydrogen Bonds in Enzyme Functions

The foregoing result has often been reported in special features on protein engineering. However, principles necessary for protein engineering will not

Table 1. Comparison of Enzyme Activity $K_m$, $k_{cat}$ of Variant Enzymes

| Enzymes | Tyrosine activation | | | Aminoacylation | |
|---|---|---|---|---|---|
| | $k_{cat}$ ($s^{-1}$) | $K_m$ (ATP) (mM) | $K_m$ (Tyr) ($\mu$M) | $k_{cat}$ ($s^{-1}$) | $K_m$ (ATP) (mM) |
| Wild type | 8.35 | 1.08 | 2.23 | 4.7 | 2.5 |
| Tyr→Phe 34 | 6.86 | 1.2 | 4.4 | — | — |
| Cys→Gly 35 | 2.95 | 2.6 | 2.7 | 1.9 | 6.1 |
| Cys→Ser 35 | 2.52 | 2.4 | 2.6 | — | — |
| His→Asn 48 | 7.90 | 1.4 | 3.8 | 4.9 | 2.1 |
| His→Gly 48 | 2.00 | 1.3 | 3.2 | 2.4 | 8.7 |
| Thr→Ala 51 | 8.75 | 0.54 | 2.0 | 4.0 | 1.25 |
| Thr→Pro 51 | 12.4 | 0.058 | 1.7 | 1.8 | 0.019 |
| Tyr→Phe169 | 6.05 | 1.25 | 10.0 | — | — |
| Gln→Gly195 | 0.19 | 2.5 | 100 | — | — |

be discovered solely by substituting amino acids and looking into the activity of the variant enzymes.

Recently, a group led by Fersht interpreted the enzyme activity of many variants in a comprehensive manner, and made a quantitative study of the role of hydrogen bonds in substrate recognition of catalytic activity by the enzyme. The results of the study are introduced below.

3.1 The Role of Hydrogen Bonds in Substrate Recognition

The group made side chains smaller or changed the kind of functions concerned with hydrogen bonds by substituting Tyr 169, which is assumed to have hydrogen bonds with the amino radical of substrate tyrosine, and Gln 195 which is assumed to have a hydrogen bond with the carboxyl radical of substrate tyrosine, and the foregoing Tyr 34, Cys 35, His 48, and Thr 51. Thus, $K_m$, $k_{cat}$ of amino acid activation reactions were determined (Table 1). The change in free energy resulting from bonding of substrates can be calculated by using $K_m$, $k_{cat}$. Based on the comparison, the role played by hydrogen bonds between the substrates and enzymes in substrate recognition can be discussed in quantitative terms.

The results are as follows:

(1) If hydrogen bonds without substrate charges are removed by substituting the amino acid side chains of enzymes (Thr 34, Cys 35, His 48), the bonds between the substrates and the enzymes are weakened by 0.5 - 1.5 kcal $mol^{-1}$. This means hydrogen bonds without charges make a comparatively small contribution to substrate recognition, boosting the recognition ability by only 2.5-15 times.

(2) If hydroben bonds with substrate charges are removed (Tyr 169, Gln 195) in the same way, bonds between the substrates and the enzymes are weakened by about 4 kcal $mol^{-1}$. This means hydrogen bonds with charges make a considerable contribution to substrate recognition, boosting the recognition ability by as much as 1,000 times.

(3) If specially weak hydrogen bonds whose distance or angle is inappropriate (Thr 51) are removed, recognition of substrates can be improved.

The above knowledge provides us with concrete guidelines when trying to alter the substrate specificity of enzymes by protein engineering. For instance, it is understood that an OH radical of the side chain of substrate tyrosine is recognized mainly by Asp 176, and the contribution of Tyr 34 is small. Accumulation of data will enable quantitative estimations of specificity increases or decreases as a result of removing specific hydrogen bonds.

3.2 Classification of Hydrogen Bonds in Terms of Catalytic Activity of Enzymes

In this chapter, the validity of applying the protein engineering method to an enzyme's catalytic power will be explained. In order to ensure thermodynamic accuracy, substrate dissociation constant Ks and speed constant k will be used hereafter in place of $K_m$ and $k_{cat}$, respectively. Their values can be determined through equilibrium dialysis and stopped flow experiments.

For a long time many approaches have been made to explain the high catalytic power of enzymes. One argument attributes the increased reaction speed to the existence of enzyme functions which have higher mutual reaction with substrates in the transition state than substrates in the ground state, and with the resultant lowering of reaction activity energy. Accordingly, enzyme functions that have mutual reactions with substrates can be divided into three groups.

(1) It is understood that enzyme functions which mutually react to substrates in the ground and in the transition state to the same extent are concerned with substrate recognition. Removal of these functions increases Ks, but does not change k.

(2) Enzyme functions which have mutual reactions only with substrates in the transition state are presumed to be concerned with the manifestation of the catalytic power of enzymes. Removal of these functions decrease k, but does not change $K_s$.

(3) Combined type of (1) and (2). Removal of these functions increases $K_s$ and decreases k. From the fact that the removal of OH radicals from Tyr 34 and Tyr 169 increases $K_s$ for substrate tyrosine, while keeping k unchanged, it is understood those OH radicals of tyrosine recognize the Oh radicals of substrate tyrosine and amino radicals (Table 2). On the other hand, removal of OH radicals from Cys 35 decreases k by one place, but hardly changes $K_s$. This means OH radicals of Cys 35 contribute to the lowering of the activation energy in the amino acid activation reaction. Removal of imidazole radicals of His 48 shows the characteristics of the combined type, decreasing k and

| Enzymes | $k$ ($s^{-1}$) | $K_s$ (ATP) (mM) | $K_s$(Tyr) ($\mu$M) |
|---|---|---|---|
| Wild type | 38 | 4.7 | 12 |
| Tyr→Phe 34 | 35 | 4.4 | 29 |
| Cys→Gly 35 | 4.0 | 4.5 | 11 |
| Cys→Ser 35 | 4.7 | 4.8 | 8 |
| His→Gly 48 | 9.9 | 9.9 | 23 |
| Thr→Ala 51 | 75 | 4.7 | 12 |
| Tyr→Phe169 | 35 | 4.6 | 1.320 |

Table 2. Comparison of Dissociation Constant $K_s$ and Reaction Speed Constant k in Amino Acid Activation of Variant Enzymes

increasing $K_s$ for ATP at the same time. Removal of OH radicals from Thr 51 increases k, but does not change $K_s$ for ATP. This is the reverse of type 2.

Classification of enzyme functions mutually reacting with substrates is useful for protein designing. This is the first step toward designing proteins so as to improve their catalytic power without changing the specificity to substrates or to tighten or relax the substrate specificity. This type of information about enzymes is useful for drug designing also, which is designing technology of substrates. It should enable highly logical designs of enzyme specific inhibitors.

4. Enzyme Activity Control

Control of enzyme activity is an important issue in protein engineering, apart from specificity alternation of substrates and improvement of catalytic power. The group of Winter and Fersht approached this issue.

It was already mentioned that Tyr RS is a dimer. It is known that two subunits of TyrRS exhibit asymmetry in terms of substrate bonds and reactivity. In other words, TyrRS bonds to only 1 mol of tyrosine or TRNA, and synthesizes only 1 mol of tyrosine adenylic acid. As the asymmetry is presumed to result from the mutual reaction between the two subunits, there is a possibility of controlling enzyme activity by the substitution amino acids located on the contact surface of the subunits.

Phe 164 was selected as the first target, because it is located on the symmetrical two-fold axis of TyrRS, and two Phe 164 adjoin each other. Substituting Phe with Asp was expected to raise the pH, and a higher pH was presumed to dissociate the carboxyl radical of Asp and cause electrostatic repulsion and dissociation into monomers. Actually, when the pH was raised from 6.0 to 7.8, dissociation to monomers and resultant loss of amino acid activation activity were observed. As the next goal, production of transformant like Phe → Lys 164 is planned to produce hybrid dimers with Asp 164 transformant. This will provide an effective means of further analysis, enabling combinations of different kinds of amino acid substituted subunits.

In an attempt to control the pH dependency of enzyme activity, Fersht's group changed the charges of amino acid residues located on enzyme surfaces, and analyzed the effect. The group used subtilisin, a kind of serine-protease, as the enzyme.

When Asp 99, located 14-15 Å away from an active part, was substituted with Ser, pKa of subtilisin enzyme activity changed from 7.17 to 6.88. The decrease in the enzyme activity was only about 80 percent. However, it is expected that substitution of nearer amino acid residues or simultaneous substitution of multiple residues will make a substantial change in the pH dependency of enzymes. When relevant data are accumulated in the future, it will be possible to determine which amino acid should be substituted in order to make a certain change in pKa.

5. Problems of Amino Acid Substitution

One of the common problems is the difficulty of estimating to what extent the three-dimensional structure of a protein is changed by amino acid substitution. Concerned with this problem, Fersht's group minimized the possibility of major structural change in proteins by limiting amino acid substitution to only one residue and restricting the direction of the change to smaller side chains. They studied a large number of cases and argued that by proving there is no contradiction among the results of the cases, they can show that change in enzyme activity results directly from the change in amino acid side chains. They suggested a method to verify whether a major change in protein structure took place, based on analysis of the activity speed.

However, we consider it essential to monitor, in one physicochemical way or another, changes in the three-dimensional structure of proteins caused by amino acid substitution. What is also important is to feed back the structural information to the next protein engineered.

X-ray crystal analysis is one of the physicochemical methods that can be used. But it may be hard to develop a routine from it, as crystallization is difficult and time consuming. Nuclear magnetic resonance (NMR) could be an alternative to X-ray analysis. However, it has rarely been used as an effective analytical means of protein engineering. Its effective use is yet to be developed.

Conformation Analysis of Low Molecular Ligand Bonded to Enzymes by New NMR

A new method of NMR which is considered to be very useful for future protein engineering or drug designing is discussed below.

The authors have established methodology to analyze conformation of amino acids bonded to ARS in detail by applying a new method, transferred nuclear Overhauser effect (acronym is TRNOE). Figure 3 illustrates the principle of TRNOE.

Low molecular ligand such as a nucleotide is assumed to shift from a high molecular-bonded state to the free state and vice versa (chemical exchange).

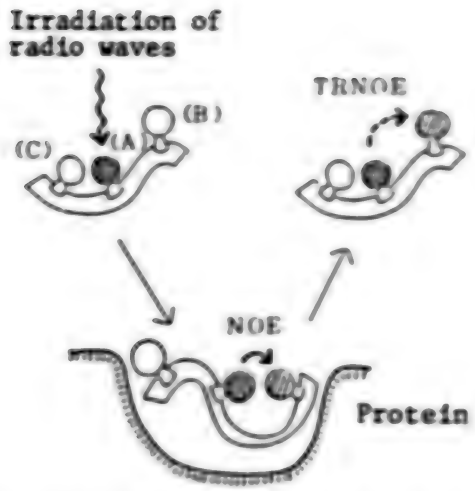


Figure 3. Principle of Bond Conformation Analysis of Low Molecular Ligand by Transferred Nuclear Overhauser Effect (TRNOE)

The proton signal of a ligand bonded to protein is too broad to be detected, whilethat of a free ligand is slightly broad (due to a change to bonded state), but still clearly observed. Radiowaves are applied to a proton (A) of a free ligand molecule. When this ligand molecule shifts to the bonded state, saturation is carried to another proton (B) near proton A. When it returns to the free state, proton B still remains saturated, and the resonance signal is weakened. By observing TRNOE, the conformation of a ligand bonded to a protein with large molecular weight can also be analyzed.

The conformation of L-isoleucyn bonded to colon bacillus isoleucyl tRNA synthetase (IleRS) (molecular weight: 110,000) was analyzed with this method. There are nine possible conformations of L-isoleucyn side chains. Figure 4 shows the conformation when L-isoleucyn is bonded to LkeRS (the side chain is extended vertically). This conformation, determined solely through TRNOE analysis, is stable and does not have steric hindrance. Conformation determination of L-valine, L-alloisoleucyn, and LleRS-bonded furanomycin, an antibiotic, in addition to L-isoleucyn was positive (Figure 4).

Bond conformation of substrates or substrate analogues determined by TRNOE can be used as a standard when monitoring changes in protein structure caused by amino acid residue substitution. Furthermore, TRNOE will provide important information for drug designing and serve as an essential method of protein engineering.

6. Conclusion

We have discussed research that could help discover principles of protein engineering, focusing on the work by a group led by Winter and Fersht. Many excellent studies have been made by other researchers as well. However, this group took the lead in terms of the data volume and systematic interpretation. Further research will make it possible to take a highly logical approach to changing proteins.

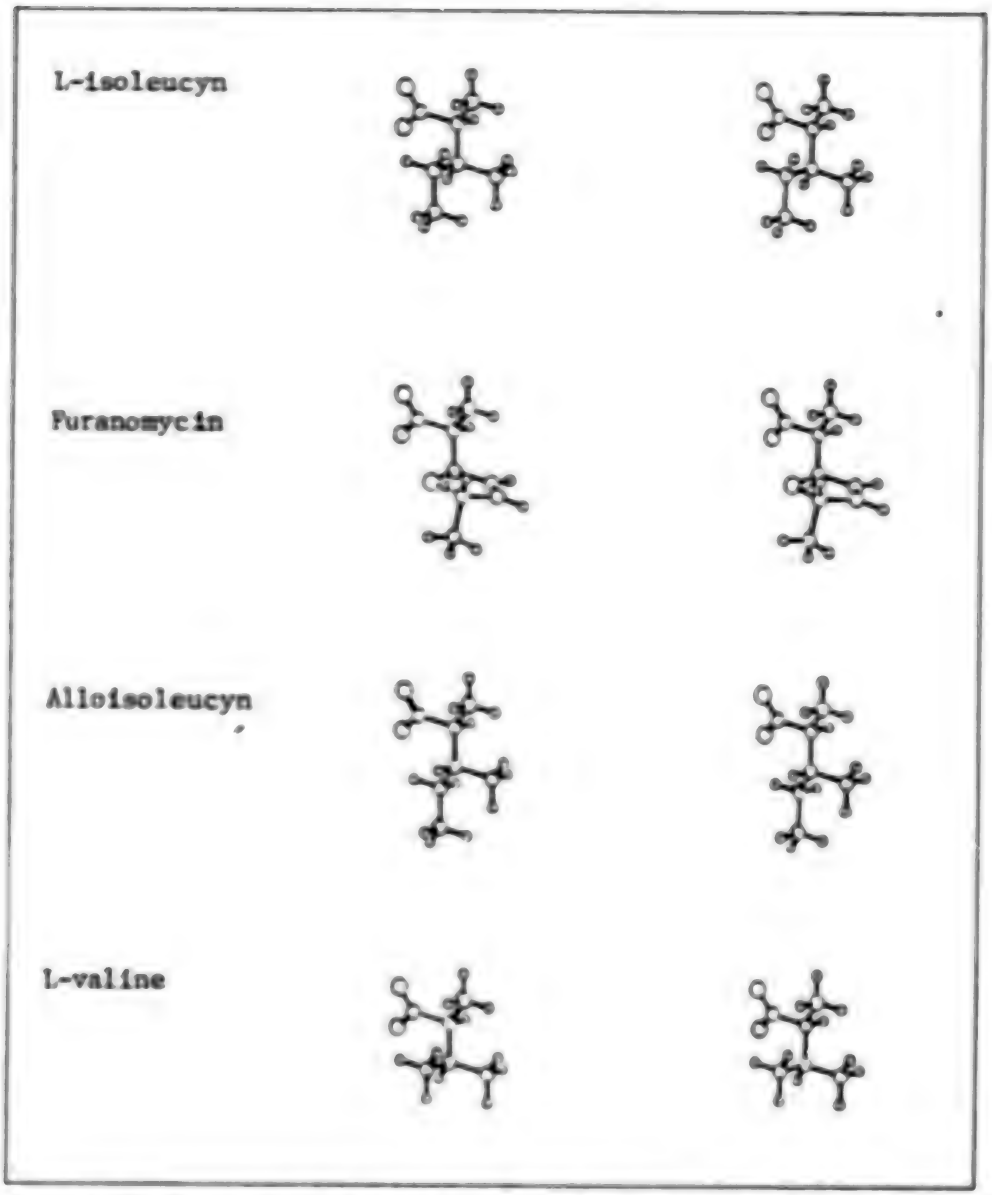


Figure 4. Conformations of L-isoleucyn and Its Analogues When They Are Bonded to Colon Bacillus Isoleucyl tRNA Synthetase

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## JAPANESE BIOTECH VIA BIJANCA DATABASE

Frankfurt/Main FRANKFURTER ZEITUNG/BLICK DURCH DIE WIRTSCHAFT in German 5 Feb 87 p 7

[Text] German enterprises and research facilities in the future will have direct access to current information concerning new developments in biotechnology in more than 2500 Japanese research facilities. This will be accomplished through the new database Bijanca at the Association for Biotechnological Research mbH (GBF) in Braunschweig. The database will be available at the end of 1987 for research jobs. According to the estimate of the Braunschweig Association, Japan, now as before, is the world leader in classical bioengineering processes such as are used, for example, in manufacturing alcohol on a large scale. In the area of modern bioengineering (gene technology and molecular biology), Japanese science and business, just like the Federal Republic, have been trying for about 4 years to catch up with the Americans. The world market value of all bioengineering processes is estimated by the GBF at about 250 billion dollars.

At the present time, according to the observations of the Federal Research Ministry, the Japanese are making considerable efforts to become stronger in modern bioengineering. According to the estimates of the Japanese biotechnology development corporation (Bidec), the proportion of biotechnologies in the gross social product will presumably increase from the present 4 percent to 10 percent by the year 2000 (approximately 200 billion DM). Approximately 200 large and medium businesses from pharmacology, chemistry, and the foodstuff industry have already for years been preparing the construction of biotechnical business branches or have already put new products on the market. Even the Japanese steel industry and the breweries are intensely interested in biotechnology.

The Japanese Foreign Trade Ministry Miti presented a corresponding "Technopolis Concept" for 18 developmental areas, already in 1980. Furthermore, in the meantime, 36 of the 46 prefectures have established biotechnical development associations to prepare the local medium and small industry for the new working methods. Improvements in fruit, vegetable, and flower cultivation by biotechnically derived, virus-free seed material as well as improvements in the embryo transfer technique for quality enhancement of meat and egg production, and new methods of fish production are

being emphasized, for instance by feeding the stock with growth hormones produced by gene-technological methods.

According to the data from the Federal Research Ministry, 129 businesses were already on the market in 1985 in Japan with new biotechnical products, as compared with only 18 businesses in the Federal Republic. According to an inquiry by the parliamentary Enquete Commission "Opportunities and Risks in Gene Technology", about 30 businesses were working on gene-technological processes and products in the Federal Republic (end of 1984). According to the representations of the commission, especially Bayer AG (blood coagulation factors), Bioferon (gamma interferon), Biotest (lymphokine), Boehringer, Mannheim (monoclonal antibodies), Hoechst AG (insulin, interferons, interleukines), Boehringer Ingelheim (TPA), BASF (TNF), were particularly concerned with gene-technological product developments.

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LASER TECHNOLOGY

## APPLICATION TO SEMICONDUCTOR PRODUCTION DISCUSSED

Tokyo OPTRONICS in Japanese Oct 86 pp 60-63

[Article by Shigeru Morikawa of Ishikawajima-Harima Heavy Industries Co., Ltd.]

[Excerpt] 2. Trend of the Technology for Processing Semiconductors Noted in the Reports Made at the CLEO Meeting

2.1 Laser CVD

Table 1 presents a summary of the reports on the laser CVD method. As can be seen from the table, the method involves use of multifarious materials including the metals Zn, Te, Cu, etc., the insulating materials SiNx and AlON, and the alloys Al-Zn and Mo-Ni. Lasers of various types are also selected in accordance with the variation of the material used, e.g., excimer laser (KrF, ArF), $Co_2$ laser, $Ar^+$ laser, and YAG excited pigment lasers, which results in combinations of a fairly large number. The author presents these reports in two categories, reaction analysis and manufacture, because space limitations prevent a detailed description of individual reports.

(1) Mechanism of Laser CVD

(i) The CVD method is contributing heavily to the development of planar technology. The laser CVD method, in particular, has applications in the manufacture of surface protecting films such as oxide- and nitride-films. There are further applications to the silicide- and oxide-films, that have elements of the III and V groups admixed; the method has become the focus of attention of researchers as a device which accomplishes reaction at low temperatures and provides products free of stress and of high homogeneity.

The CVD method provides materials of multifarious properties because it allows selection of relevant parameters such as temperature, gas composition, concentration, and pressure. It, nevertheless, involves unpredictable factors heavily and the mechanism of its reaction remains largely unknown.

With the view to elucidating the mechanism involved, Chen set forward the following CVD formula on the basis of the kinetic theory and tested on ZN for accuracy.

Table 1. Summary of the Reports on Laser CVD

| Research Organization | Reporter | Objective or Subject of Research | Types of Lasers Used | Reaction or Process Conditions, etc. | Reference |
|---|---|---|---|---|---|
| IBM | C. J. Chen | Investigation of the mechanism of the laser CVD method by setting up a theoretical formula, which the researcher verified by CVD experiments with Zn | | Photochemical decomposition of diethyl zinc dissociation of absorbed substances at room temperature and predominantly photo-dissociation in gas phase at high temperatures are involved | |
| IBM | J. M. Jasinski | Investigation of the mechanism of the laser CVD method, investigation of the formation of $SiH_2$ from $SiH_4$ | TEA-$CO_2$ excimer laser, CW-pigment laser | Formation of $SiH_2$ with the two lasers and monitoring of $SiH_2$ with CW-pigment laser | |
| Max Planck Institute | R. Larciprete et al. | Investigation of the mechanism of the MOCVD method; application of laser mass analys | Excimer laser | Determination of optimum conditions (wavelength intensity); application of the CVD method to $CH_3TeCH_3$, $CH_3TeTeCH_3$, $C_2H_5TeC_2H_5$, TIBA, etc. | Application of pico-seconds laser |
| Colorado State University | T. Y. Sheng, et al. | Investigation of AlON with the view to enhancing the electrical performance of the Al/AlON/InP diode | ArF (193 nm) excimer laser | Specimen subjected to cleansing with 1 percent Br-methanol; baking in vacuum ($10^{-6}$ Torr) at 60 degrees C for 6 hours; evacuation of air for 15 hours, $NH_3/N_2$=50/600 sccm; temperature of substrate at 300 degrees C, $NH_3$/trimethyl ammonium=50/0.1 | Measurement of film thickness with ellipsometers, 800 Å/18 minutes |
| Amoco | K. V. Reddy | Investigation of the CVD method for crystal silicon, amorphous silicon and copper concerning the effect of Ar and other inert gases on the deposition speed | KrF excimer laser | A coating of Ar applied to the Si substrate at 15 degrees K with subsequent CVD using amorphous Si or Cu | |
| Ishikawajima Harima Heavy Industries Co., Ltd. | S. Morikawa | Investigation of the mechanism of the CVD method for $Si_3N_4$ | YAG-pigment laser | $NH_3$-$SiH_4$ system, $NH_3$-$Si_2H_6$ system, 1-40 Torr, substrate temperature at 250-300 degrees C; wavelength 196-530 nanometers | |
| AT&T Bell Lab. | G. S. Higashi, et al. | CVD for Al, Si, GaAs, etc. | KrF (248 nanometers) excimer laser (100 Hz) | Triisobuthyl aluminium/$H_2$ as the raw materials; reaction temperature of 250 degrees C; magnification of the microscope X15 used for narrowing laser intensity 20mJ/cm$^2$ | The deposition speed is influenced by the partial pressures of the material gases |
| Sperry Corp. | R. J. Baseman, et al. | CVD of the alloy Al-Zn | $Ar^+$ (257 nanometers) laser | Dimethyl-zinc-trimethyl — aluminium as the raw material; under a pressure of 2.2 Torr | The plot of the deposition speed versus the partial pressure of dimethyl zinc showing a maximum value |
| Los Alamos National Lab. | T. B. Jervis | CVD of the alloy Mo-Si | $CO_2$ laser | Raw material: $Si(CO)_4$/$MoF_6$/$H_2$/Ar | Application of TEM and Auger electron spectroscopy for the assessment of films |
| Colorado State University | M. Zarnani, et al. | CVD of amorphous Si:H | ArF (193 nanometers) laser | $Si_2H_6$/He as the raw material; pressure of 1-10 Torr; the angle of the laser beam ranging from 0 degrees to 90 degrees; the temperature of the substrate at 35-350 degrees C | Application of FT-IR and XPS for the assessment of the film |

$$T(y)=\frac{CE\sigma_0}{\left(1-\frac{P}{P_0}\right)\left[1-(C-1)\frac{P}{P_0}\right]}\times\frac{\mu S_0}{\sqrt{\pi}V_0N_0}\exp\left(-\frac{y^2}{a^2}\right) \quad (1)$$

where T(y) stands for the thickness of the layer deposited, C the constant of the BET formula, E the rate of increase, $\sigma_0$ the cross-section of photodissociation of the reacting gas, P the partial pressure of the reacting gas, Po the vapor pressure of the reacting gas, $\mu$ the molecular weight of the product, So number of photons, V scanning speed of the laser beam, a the radius of the laser beam, N the Avogadro number, and $\rho$ the density of the products. Chen's test on Zn for CVD has proved that the formula is tenable, but different elements need to be tested for CVD and the applicability of the formula to them need to be ensured if the formula is to be used efficiently. The author quoted particularly the above formula because of the recent tendency for the elucidation of the mechanism to give excessive weight to physical properties and characterization of substances and rarely to dwell on the kinetic theory. If a general rule based on the above formula is set up, it, along with characterization of substances, may be able to lead to the development of laser CVD.

ii) Morikawa, et al., inferred the mechanism for the formation of silicon nitride films, using lasers of variable wavelength: They investigated SiNx produced by the reaction of $NH_3$ with $SiH_4$ or $Si_2H_6$ in the wavelength range of 190-530 nonameters and have found that the formation of the film is possible in the wavelength range above 230 nonameters where the absorption for $SiH_4$ and $Si_2H_6$ is lacking but ones for $NH_3$ are present. It had been believed till then in connection with the selection of wavelengths for CVD that the formation of that film is possible exclusively in the range of absorption spectra for $SiH_4$ and $Si_2H_6$. They thus concluded that the reaction of $SiH_4$ and $Si_2H_6$ with $NH_3$ proceeds by way of the excitation of $NH_3$ and the formation of a relevant activated complex. A comprehensive application of lasers of variable wavelength thus has elucidated the wavelength characteristics and suggested the presence, for laser reactions, of an optimum wavelength other than those which have to date been used in lasers of single, fixed wavelengths. SiNx films produced in this way compared with those produced by means of the thermal CVD in the uniformity of the film, step coverage, etc.

(2) Manufacture of Films by Means of Laser CVD

(i) CVD of alloy: In addition to its advantage of affording a uniform thickness and quality of the film and allowing reactions to take place at low temperature, the laser CVD is also favored by its being capable of forming metal film at any particular site on the substrate. The formation of metal films by this means, meanwhile, is possible for almost all metals by using raw material compounds such as hydrides, halides, and organic metal compounds which are subjected to reduction or decomposition to give films.

Baseman, Jervis, et al., meanwhile, reported on the CVD of the alloys Al-Zn and Mo-Ni, of which the raw materials are organic metals and carbonyl compounds as in the case of single metals. In the case of the alloy Mo-Ni, the formation of its films is affected by the relevant pressure, i.e.,

excessive pressures lead to the formation of a powder and a metastable amorphous product comparable to the formation of a superfine powder by the application of $CO_2$-laser. The alloy Al-Zn, in turn, is formed into film by means of harmonics of $Ar^+$-laser. Though such metals as Al and Cd have been formed into film to date by means of photodissociation of their alkyl metals with the above $Ar^+$ laser, the partial pressures of raw materials seems to affect the speed of film formation when alloy systems are used in place of the homogeneous system of single metals. Their report lacks a comprehensive analysis of the parameters dictating the reaction and leaves many factors, including the quality, homogeneity, etc. of the film unclarified. Demands for alloy film, nevertheless, seem to rise with growing demands for metal films in future in connection with the demands for silicides, ohmic contacts, mask correction, etc., which is the reason for dealing with the above two alloys here.

ii) Insulation films: Sheng investigated AlON as an insulation film for the device MIS (Metal-Insulator-Semiconductor). This material has been spotlighted as a new type of insulation film in contrast to those of $SiO_2$, SiNx, PSG, and BSG which have so far been available. Chen, in a report, pointed up insulators as responsible for the electrical instability of the device and the current drift of the diode of Al-AlON-InP and demonstrated, by means of IMHz-C-V-characteristics changes, that the above shortcoming can be eliminated by, among other things, reducing damages to the surface of the AlON.

iii) Enhanced speed of film formation: In order to increase the speed for the formation of the film of amorphous Cu and Si, Reedy applied a coating to the substrate with an inert gas such as Ar, $N_2$, or Kr at a very low temperature of 15 degrees K with subsequent CVD and found that the threshold level of laser intensity required for the film formation went down from 4.0 $J/cm^2$ to 2.5 $J/cm^2$ in the case of Ar coating and that the speed of film formation grew around three times.

It is not yet known whether the role of inert gases Ar, $N_2$, etc. is equivalent to that of the photosensitizer in the photoexcitation method, but it is hoped that reports of this kind increase in number in the coming years.

2-2. Laser Etching and Others

A summary of laser etching is presented in Table 2 and that of laser annealing, direct writing, and doping, in Table 3.

In connection with laser etching, J. H. Bannon carried out etching of Si of 1.5 microns with $NF_3$ under exposure to $CO_2$-laser and found that the speed of etching varied with wavenumber ($cm^{-1}$) and that, of a number of absorption lines, the wave number 922.85 $cm^{-1}$ was effective. Whereas $Ar^+$ laser and excimer laser, of all types of lasers, have been frequently used to date for etching, the current meeting focused attention on the use of UV laser of variable wavelength reported by J. Alspector as well as on the use of $CO_2$-laser described above.

Table 2. Summary of Reports on the Laser Etching

| Research Organ | Reporters | Objective and Subject of Researches | Lasers Used | Reaction or Process Conditions | Reference |
|---|---|---|---|---|---|
| IBM | R. W. Dreyfus, et al. | Investigation of the mechanism of etching for $Al_2O_3$ | Excimer laser, 1.2-1.5 J/cm$^2$ | Density of particles and changes in physical states elucidated by a comprehensive application of the measurement of laser induced fluorescence (LIF) | The degree of etching dictated by the $O_2$ surface-coating |
| Bell Communication Research | J. Alspector | Investigation of a damage-free etching of Al; application in IC's being planned | UV laser of variable wavelength | Wavelength of 3,550-4,000 Å; specimens of 0.4-1.4 microns in thickness and 1.0-1.25 microns in width; and 90 percent $HNO_3$/10 percent $H_3PO_4$/0.15 percent by weight of $K_2Cr_2O_7$ used as the raw materials; microscope (object lense X50, X100) used | Compare with the experimental results of Ehrlich, et al. |
| IBM | J. M. Bannon | Investigation of the etching of SI with $NF_3$ | $CO_2$ laser | Involving a reaction between Si and F, the latter liberated by the decomposition of $NF_3$; $NF_3$ at a pressure of 150 Torr ($BaF_2$ window plate, collection of light with ZnSe lense) luminescence line of 922.85 cm$^{-1}$ used | Application of a quartz microbalance for the determination of the rate of etching; Si-1.5 micron etching |
| Columbia University | A. D. V. Podlesnik | Both experimental and theoretical investigation of the etching of InP | Ar laser (257 nm) | Laser intensity (50-3,000 watts/cm$^2$) spot size 3 microns; etching with an HF solution | |
| Israel Institute of Technology | G. Koren | Investigation of the etching of Mo and W under the atmosphere | $Ar^+$ laser (257 nm) | 50-micron Mo-foils and 150-micron W-foils used as the specimens; laser intensity of 3-13 watts | Measurement of grooves and holes with SEM |

1. D. J. Ehrlich, et al., IEE Trans. Electron Devices Lett. EDL-5, 32 (1984).

Table 3. Summary of Reports on Laser Annealing

| Research Organ | Reporters | Research Objective and Subject | Laser Used | Reaction or Process Condition | Reference |
|---|---|---|---|---|---|
| Defence Science Center | A. L. Dawar | Investigation of changes in structure and electrical performance produced in SnTe films by means of laser annealing | ND-YAG (20 nsec) (1.06 $\mu$) | Substrates of glass, mica, and KBr The temperature of substrate at 250 degrees C; the pressure of $5 \times 10^{-6}$ Torr; laser intensity of 2-50 $mJ/cm^2$ | The size of grains is affected by the laser pulse. Combined use of TEM and X-ray diffraction |
| Stanford Electronics Lab. | P. G. Carey, et al. | Formation of p- and n-junction by means of doping effected in surface layers of Si | XeCl (308 nm) excimer laser | Leakage current, $I_D$-$V_G$ characteristics seen; annealing, in case it is applied, is at 950 degrees C for 10 seconds; changes due to application of annealing also examined | Extent of doping checked with SIMS |
| MIT | J. G. Black, et al. | Investigation of the relationship between the laser direct writing and the deposition process | Excimer laser | $B_2H_6$-doped Si and W used in deposition as the raw material | Writing speed of 2.5 mm/s |

The method of etching involved is not particularly different from those so far used. The laser beam of wavelength variable in the range from 3,550 to 4,000 Å, when tested, pointed up wavelength 3,973 Å as adapted to Al etching, a wavelength which allows fine etching of Al to be made with less damage and which may possibly be applied to MOS devices. As can be seen from the above J. Alspector's case, laser etching seems to shift its weight from thermal lasers and plasmic lasers of short wavelength to lasers of wavelength range wherein photochemical reactions prevail.

Reports on laser annealing, laser doping, and laser direct writing did not particularly attract the author's attention and hence are not referred to here.

3. Conclusion

As described at the beginning, some 700 reports were made at the current CLEO and the report on the semiconductor processing technology presented above, of necessity, cannot cover all of the relevant reports. The author fears that the selection of topics may have been affected substantially by his own personal interest and that some important topics may have been overlooked; if so, he begs the reader's pardon.

The substrates used are mainly 6 inches in size according to the reports of the current meeting; it will not be long before the 8 inch ones are marketed.

Whereas the author thought that reports of original research would hardly be released at the CLEO since it is held annually unlike other international conferences, some reports were intriguing to the participants whether they are on oscillators or on laser applications. It was, nevertheless, regrettable that Japan, a nation with most advanced technology in semiconductor processing and in the application area of semiconductor manufacture, made only a limited number of reports.

20,128/9599
CSO: 4306/2561

## SHIPBUILDERS IMPLEMENT SUBSEA TECHNOLOGIES

Tokyo SEA-JAPAN in English Nov 86 pp 1-2

[Text]

Japan's extensive 200-mile exclusive zones of 4.5 million $km^2$ are approximately 12 times larger than land and are fraught with unexplored resources.

Much more wider R&D activities worldwide should be committed to ocean development projects in the future and in this connection, Japanese major shipbuilders have already been continuously implementing their accumulated marine and ship-related technologies in this field during these years.

Especially, great efforts are being spent on the R & D of the unmanned and the manned equipment such as the subsea vehicle, submersible inspecting robots and deepsea sources surveyers.

Introduced here are the small and lightweight type subsea equipment developed by Japanese shipbuilders to meet the users' high demand for various underwater inspections and observations.

A large market is forecast from now on as all of these equipment feature versatile usage and easy operation and moreover, subsea conditions can be observed clearly through the TV cameras.

* * *

### MHI ready to construct 2,500m-diving vehicle

Mitsubishi Heavy Industries, Ltd. (MHI) recently received an order from Kokusai Denshin Denwa Co., Ltd. (KDD) of Tokyo for an unmanned subsea vehicle, "Shinkai Marcas," which can conduct various inspection works at a sea depth of about 2,500m.

KDD had already put into practical use the unmanned vehicle, "Marcas," with a diving capacity of about 200m deep in 1981 and this time, KDD commissioned MHI to develop and manufacture a deepsea

vehicle to assist KDD's cable laying project in the Pacific Ocean.

With an achievement of completing the manned type vehicle, "Shinkai 2000," MHI had already received an order from the Agency of Science and Technology for the basic designing of "Shinkai 6000" which can dive down to a maximum depth of 6,500m.

MHI is scheduled to deliver the new "Shinkai Marcas" to KDD by April 1987; it will have a camera and sensor and will inspect the condition of the sea bottom before the cable layer places the cable or observe the condition after the cable was laid, thus, improving the efficiency of maintenance and checking jobs.

Furthermore, if an elaborate manipulator is developed, the vehicle will be able to drill the seabed, lay the cable and bury the cable into the seabed.

### 2nd unmanned subsea vehicle built by SHI

Sumitomo Heavy Industries, Ltd. (SHI) delivered the second "Hornet 500," an unmanned subsea vehicle, to the Japan Marine Science and Technology Center following the completion of the first "Hornet 500" in 1984.

The vehicle can dive down to a depth of 500m by the remotely controlled thruster from the support ship.

It consists of two spherical chambers made of Al alloy connected with each other and its shape looks like a hornet. A color TV camera and a monochrome TV camera are installed in the fore and the aft spherical chambers of the vehicle, respectively, to observe the deep sea bottom by a monitor set up on the support ship.

The optical fiber cable is used to communicate between the vehicle and the support ship.

Main particulars are:
Overall length: 120m
Overall width: 96m
Height: 56cm
Weight: abt. 130 kg.

### 3-yr. ROV program commenced by KHI

Beginning this year, Kawasaki Heavy Industries, Ltd. (KHI) started a three-year program to develop the new generation remotely operated vehicle (ROV) which is highly capable of keeping the position automatically and accurately moving in the water against the current force or the manipulating reaction force.

The new ROV will incorporate the following equipment:
(1) Three-dimensional automatic maneuvering system consisting of three kinds of sensors for depth, speed and angle; the system can control the vehicle's position exactly as previously fixed by incessantly communicating with the minicomputer equipped on the ship.
(2) Tether cable composed of optical fibers and electric power lines.
(3) Master/slave force feedback type manipulator.

In the first year of the program, KHI intends to develop the operational and controling equipment which will be installed on the ship to conduct simulation operation.

### MES sells self-propelling underwater TV cameras

A self-propelling underwater TV camera system which was developed and put on the market in April 1985 by Mitsui Engineering & Shipbuilding Co. Ltd. (MES) is now showing good results.

The underwater TV Robo Mitsui RTV-100 can dive down to a depth of 100m at a speed of two knots to observe, inspect marine, coastal, fisheries resources developments including various kinds of exploratory activities.

The TV Robo consists of the body weighing about 25kg, a control system incorporating power supply, joystick assembly, a 10-inch color TV monitor and an underwater cable system.

Main particulars are:
Overall length : 75 cm
Overall width : 55 cm
Height : 35 cm
Color TV camera
Underwater illumination lamps
Depth gauge
Bearing indicator
Underwater cable: 100m-long composite power-optical fiber cable

**Hitachi Zosen develops two underwater devices**

Hitachi Zosen Corp. has developed two kinds of underwater observation devices — Eye-Ball and Hi-ROV•15 — and started marketing them in cooperation with the National Federation of Fishers Co-operative Associations.

**Eye-Ball:**

The versatile underwater probing and observation device has a color TV camera protected by a clear, waterproof, synthetic resin spherical shield.

It has a wide range of applications which include artificial reef, fishing banks and fishing net locations, dock and harbor facilities, dam gates and recreation purposes.

The camera, equipped with tilting and panning gears, is operated by remote control via an electric cable.

The electric power source is AC 100 volts (with lights) or DC 12 volts (without lights).

Main characteristics are:
Operating depth: 100 m max.
Globe diameter: 220 mm
Weight: 5 kg
Camera pan/tilt: Pan 360 degrees; tilt 230 degrees

**HI-ROV•15:**

The HI-ROV•15 is an easy-to-use, mobile underwater observation and exploration robot and is operable via tethered cable down to a depth of 150 meters.

It can be used for inspection, nondestructive test, sampling and other underwater tasks in a variety of industrial or recreational fields. In deep and fast current seas, two halogenous lamps provide a clear view and three powerful thrusters facilitate underwater positioning.

Main features are:
Operating depth: 150m max.
Speed: 3.3kt max.
Weight: 25kg
Body: Anodized and painted aluminum hardcoat
Thruster: 150W x 3 thrusters
Depth gauge: 0 - 150m

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CSO: 4307/514

# RECENT DEVELOPMENTS IN CERAMICS PROCESSING TECHNOLOGY DISCUSSED

Tokyo CERAMICS JAPAN in Japanese Aug 86 pp 676-682

[Article by Tadashi Hisamoto, Hisamoto Consulting Office: "Progress in Precision System Processing of Advanced Ceramics and Proposal To Design Ceramic Materials From Processing Engineering"]

[Text] 1. Introduction

There are high expectations for ceramics as building materials because of their characteristics of being hard, highly heat resistant, and chemically stable.

But compared with metals and plastics, ceramics are difficult to process, and this constitutes a big barrier in expanding their applications as building materials.

Since last year the society held seminars and symposia as shown in Tables 1-4. I would like to summarize their proceedings, as well as topics in the general meeting of the Precision Engineering Society and local lecture meetings, along with some comments of my own.

2. Objectives of Precision System Processing of Ceramics

The objective of the CP Subcommittee of the Precision Engineering Society is to tackle the difficult machinability of ceramics for building materials, develop low cost production processing technologies, and give credibility to the materials so that their scope of application can be expanded. As materials to study, we would like to include $Al_2O_3 \cdot ZrO_2$ as oxide system and SiC, $Si_3N_4$ and Sialon as nonoxide systems and also new materials such as AlN, $TiB_2$, and $ZrB_2$.

These ceramics have very large heat expansion coefficients and thus constrict substantially upon baking, making it very difficult to produce unprocessed pieces of an exact dimension. Thus, today it is necessary to perform one-shot precision processing after baking.

From the viewpoint of low-cost production, however, the cost for the one-shot precision processing should be restricted. To cope with this it has been devised, in addition to the process of precision molding and baking, to perform preliminary precision processing before baking or upon preliminary baking

Table 1. 12th PS Seminar of Precision Machine Society "Super-Precision Processing of Ceramics"
Wednesday, 6 March 1985 (Nagoya Industrial Research Institute, Agency of Industrial Science and Technology) - Thursday, 7 March 1985 (Nagoya Small and Medium Size Enterprise Promotion Hall)

| | Items | Themes | Lecturers |
|---|---|---|---|
| 6 March | 1. Greeting | Purpose of the seminar | (PS Special Committee Chairman) Hisamoto |
| | | Introduction of Nagoya Industrial Research Institute | (Director, NIRI) Nagase |
| | 2. Tour | Tour of NIRI ceramic laboratories | |
| | 3. Lecture on technologies | Superprecision system processing for fine ceramics and its objective | (Chief, CP Subcommittee) Hisamoto |
| | | Molding and baking technologies of Fine Ceramics as Preliminary process of efficient superprecision processing | (NGK Spark Plug Co., Ltd.) Shibata |
| | | Special processing of fine ceramics | (Toyota Institute of Technology) Saito |
| | | Application of ultrasonic compound processing of fine ceramics | (Ultrasonic Industry) Ishiwatari |
| | 4. Questions and discussions | Question and answer session with PS committee and CP subcommittee members | Chairman (PS Special Committee member) Kinoshita |
| 7 March | 1. Greeting | How to see '85 Ceramic Fair | (Takahiro Industry) Takagi |
| | 2. Lecture on technologies | Workability of fine ceramics as seen from functional design | (Tokyo University) Imanaka |
| | | Grinding work and evaluation of processed surface | (NIRI) Itoh |
| | 3. Tour | Tour of Fine Ceramics Fair '85 | Fukiage Exhibition Hall |
| | 4. Lunch | No 3 Conference Room | |
| | 5. Lectures on technologies | MEEC processing of fine ceramics | (Ibaraki University) Kubota |
| | | Vitrified diamond grinding wheel | (Japan Grain Institute) Okada |
| | | Ceramics grinding diamond wheel | (Toshiba) Harada |
| | | Cast iron bond diamond wheel | (Tokyo University) Nakagawa |
| | | Grindability of fine ceramics | (Osaka Diamond) Tonimori |
| | | Fine ceramics evaluation attempts using ultrasonic microscope | (Hitachi, Ltd.) Ishikawa |
| | 6. Questions and answers | Question and answer session with PS committee and CP subcommittee members | Chairman (CP Subcommittee member) Takagi |
| | 7. Closing remarks | | (PS Special Committee member) Imanaka |

Table 2. Precision Machine Society "International Symposium on Low-Cost Production Processing Technology for Fine Ceramics Parts"
Thursday, 5 December-Friday, 6 December 1985 (Lecture Hall of Koyosha Building)

| Date | Themes | Lecturers |
|---|---|---|
| 5 December | 1. Opening speech: Purpose of the symposium | (CP Subcommittee chief) Hisamoto |
| | 2. General remarks: Present status and problems in manufacturing and processing fine ceramics | (Toyama Vocational Junior College) Takasawa |
| | 3. "Manufacture of Building Ceramics"<br>Case(s) of $Al_2O_3$ ceramics production automation<br>Case(s) of $Si_3N_4$ ceramics production automation<br>Case(s) of SiC ceramics | (Chairman)(Ibutoku Institute of Technology) Takasawa<br>(NGK Spark Plug Co., Ltd.) Asano<br>(Kyocera) Nakahara<br>(SOHIO) J.W. Hinton; (Hitachi Carborundum) J.A. Coppola |
| | 4. "Confirmation of Credibility of Building Materials: Part 1"<br>Needs for confirmation of credibility and case(s) of confirmation<br>Case(s) of using ultrasonic probe<br>Case(s) of using Softex (X-ray) equipment | (Chairman) (Hitachi, Ltd.) Nukaya<br>(Toyota Central Laboratory) Kamiya<br>(Hitachi Building Machine) Nonaka<br>(Softex) Okuyama |
| | 5. "Confirmation of Credibility of Building Materials: Part 2"<br>Case(s) of using acoustic emission<br>Evaluation of precision processed surface using ultrasonic microscope | (Chairman) (Ultrasonic Industry) Ishiwatari<br>(Utsunomiya University) Takeshi<br>(Hitachi, Ltd.) Ishikawa |
| | 6. General discussion: "Selection of Fine Ceramics From Functionability" | (Chairman)(Tahara Shoeikiko) Koide |
| 6 December | 1. "Production Processing Technology: Part 1"<br>Methodology of third-dimensional curve processing<br>Cast iron diamond wheel and third-dimensional processing | (Chairman) (Ibaraki University) Kubota<br>(Hokkaido University) Saito<br>(Tokyo University) Nakagawa<br>(Toyama Industrial Technology Junior College) Uematsu<br>(Tokyo University) Suzuki |
| | Possibility of fine ceramics by special processing<br>Replacement of internal surface grinding machine for fine ceramics and experiences of development and application as seen from processing and manufacturing | (Toyota Institute of Technology) Saito<br>(Taisei Machine) Ohta |
| | 2. "Production Processing Technology: Part 2"<br>Development case(s) of ceramics grinding tools<br>Case(s) of processing using diamond grinding sheet | (Chairman) (Meiji University) Tokokawa<br>(Tokyo Institute of Technology) Yoshikawa<br>(Shibaura Institute of Technology) Shibata |
| | 3. "Conclusion and Prospect"<br>Way to low-cost production of fine ceramics | (Chairman) (Takahiro Industry) Takagi<br>(Hisamoto Consulting Office) Hisamoto |
| | 4. General discussion: "Cost Measures for Fine Ceramics" | (Chairman)(Kinoshita Consulting Office) Kinoshita |

Table 3. 15th PS Seminar of Precision Engineering Society "Technologies of Special Processing and Application Expansion for Fine Ceramics" Tuesday, 4 March 1986 (Field Visit Meeting: Mitsubishi Electric Co., Ltd., Nagoya Works; Seminar: Nagoya Small and Medium Size Enterprise Promotion Hall)-Wednesday, 5 March 1986 (Nagoya Small and Medium Size Enterprise Promotion Hall)

| | Items | Themes | Lecturers |
|---|---|---|---|
| 4 March | Field visit | Meet at Nagoya Works of Mitsubishi Electric Co., Ltd.<br>Greeting<br>Attendance: Demonstration of electrical discharge machining and laser machining of fine ceramics | (Director of MEC Nagoya Works) Machino |
| | Greeting | Purpose of seminar | (PS Special Committee Chairman) Hisamoto |
| | Lectures on technologies | 1. Prospect of special processing of fine ceramics | (Toyota Institute of Technology) Saito |
| | | 2. Electrical discharge processing of fine ceramics | (Nagoya Industrial Research Institute) Nakamura |
| | | 3. Characteristics of electrical discharge processing of conductive ceramics | (Mitsubishi Electric Co., Ltd.) Ozaki |
| | | 4. Development of fine ceramics which can be processed by electrical discharge and expectation of application expansion<br>(1) with SiC ceramics<br>(2) with $Si_3N_4$ ceramics<br>(3) with Sialon | <br>(Hitachi, Ltd.) Kosugi<br>(Sumitomo Electric Engineering Co.) Takeuchi<br>(Hitachi, Ltd.) Tanaka |
| | Questions and discussions | Question and answer session with PS Special Committee and CP Subcommittee members | Chairman (PS Special Committee member) Kinoshita |
| 5 March | Greeting on reopening | Outline of fine ceramics processing | (PS Special Committee member) Imanaka |
| | Lectures on technologies | 5. Fine ceramics processing by $CO_2$ laser | (Mitsubishi Electric Co.) Mori |
| | | 6. No distortion minute processing of fine ceramics by $CO_2$ laser | (Nihon Electric Co.) Sugishima |
| | | 7. Crackless processing by $CO_2$ and YAG laser | (Toshiba) Nagano |
| | Tour | Fine Ceramics Fair (Invitation Day) | Presentation on Venue (Takahiro Industry) Takagi |
| | Lectures on technologies | 8. Joining of fine ceramics and metals | (Tokyo University) Suga |
| | | 9. Recent trend of ceramics coating | (Ibaraki University) Nagasaka |
| | | Question and answer session with PS general discussion Special Committee and CP Subcommittee members | Chairman (Special Committee member) Imanaka |
| | Greeting on closing | | (PS Special Committee member) Watanabe |

to reduce the processing cost after baking. This method has been adopted in some areas.

3. Cutting Work of Ceramics

Historically, in the normal temperature processing machine cutting started before the others in processing metals, followed by grinding, honing, and super-finish work using tools made of solidified grit.

But when it comesto glass with its hard brittleness, the story is different. The lapping method where grit is used in powder form came first and the development of the grinding method with the wheel made by sintering the grit had to wait until after World War I. In fact, I was given by Professor Ohgoshi the subject of the glass grinding method for my thesis. I received the First Thesis Award from the Precision Machine Society in 1950 and published a book "Precision Work of Glass" from Seibundo Shinkosha in 1955.

The publication of glass cutting work was some time later: a presentation (use of sintered diamond tools) by Ishikawa, et al.,[1] of Nagoya Industrial Research Institute was made at the general meeting in Akiokudani in 1979. The application of this technology, however, has not expanded much since then.

Machine cutting of ceramics, like that of glass, started after the grinding work. Sumitomo Electric Engineering Co., Ltd.,[2] took on the research, followed by Hyogo Prefectural Institute of Industrial Research,[3] and Toshiba Tungaloy Co., Ltd.,[4] among others. Sumitomo Electric Engineering succeeded by using sintered artificial diamonds with enhanced toughness. Toshiba also used sintered polycrystal tools while Hyogo Prefectural Institute worked on the material's cuttability ($Al_2O_3 + TiO_2$).

4. Grinding Work of Ceramics

The most important aspect in ceramics precision processing is after all the grinding work. The experiences we have so far acquired in metal grinding, however, cannot be applied as they are to ceramics work.

The grinding work is a process using the grinding wheel to finish the surfaces into the desired forms such as flat plane, external and internal surfaces of a cylinder, spherical surface, and curved plane. Good results cannot be expected unless three factors--grinding wheel, grinding machine, and grinding conditions--are well prepared.

(1) Grinding Wheel

The grinding wheel is made up of grit, binder, and appropriate voids.

In grinding ceramics, usually the grinding wheel with grit from artificial diamonds is used. The fact that various brands of wheels are available from GE (United States) and DeBeers (Great Britain), is important.

Thus, it is not appropriate to use a metal grinding machine for grinding ceramics. A ceramics grinding machine should have high rigidity and should be provided with protection measures for the sliding surface.

(2) Grinding Conditions

Decisions on the grinding conditions, such as selection and control of the grinding oils, truing and dressing of the wheel surface, and setting of the grinding program, represent one of the three requirements in grinding work of ceramics. The internal surface grinding machine of Taisei Machine, for example, operates as follows:

In Taisei Machine, an electrically deposited diamond wheel is used at high speed revolutions while applying a coolant to the grinding spot under high pressure jet. Filter and freezer are built into the coolant circulation system to successfully apply cuts by the NC control.

5. Compound Grinding Work of Ceramics

(1) Ultrasonic Grinding Work

While the ultrasonic grinding using powder grit is effective for drilling glass because of its good workability, the same process cannot be easily applied to ceramics.

Lunzer (United States)[6] developed electrically deposited diamond drills (solid drill for small diameters and hollow drill for large diameters are available), and formed a system with an ultrasonic machine of Branson (United States). The marketed system, however, has the drawback of poor machine rigidity.

In Japan, Osaka Diamond and Shimada Physicochemical recently developed an ultrasonic grinder in collaboration with a vocational college.[7] Compared with the U.S. system, this system has a higher rigidity and can be controlled by NC (computer). While this can be used for ceramics processing, expansion of the application field is limited due to the one-way (vertical) vibration of the ultrasonic wave.

(2) Electrolysis, Electrical Discharge Grinding Work

Kuromatsu[8] of Applied Magnetic Research Institute successfully developed the MEEC method--a compound process of electrolysis, electrical discharge, and mechanical grinding--to process cendust (magnetic material) which is hard to machine. The commercial message that this method could be applied to ceramics processing was disseminated to make manufacturers of grinding wheels and grinding machines hurriedly introduce the MEEC method and commercialize the products. But the industrial application proved that the method (though suitable for expensive cendust and functional ceramics) is yet to be satisfactory for processing low-cost mass production ceramics to be used as building materials.

## 6. Electrical Discharge Processing of Ceramics

Pioneers in electrical discharge processing in Japan are Professors Ohgi (Electric Engineering) and Kurafuji (Precision Engineering) of Tokyo University. Incidentally, I was in the same class at Tokyo University with Professor Kurafuji and was also interested in the application of electrical discharge processing to publish a report on the electrical discharge processing of WC (superhard) dies for wires.[9]

When I opened the consulting office 9 years ago specializing in ceramics processing, electrical discharge processing, as well as machine grinding, were under consideration. When I visited and discussed with Dr Saito, who had at that time just taken up the professorship of Toyota Institute of Technology after resigning as chief engineer of Mitsubishi Electric Co., Ltd., the topic of electrical discharge processing of ceramics came up and the doctor said that electrical discharge processing was possible for SiC ceramics. The discussion took place before Sumitomo Electric Industry's publication[10] regarding the provision of electric conductivity and the issue at that time was whether available ceramics could be processed by electrical discharge. Because of such circumstances, when the CP Subcommittee started, I asked Professor Saito and Professor Kubota of Kabaraki University Faculty of Engineering for their participation.

It is extremely difficult to set boundaries as to whether electrical discharge work is possible or not because it depends on the composition of the materials and the sintering method employed, and also varies with the uneven distribution of the components. If we are to set a boundary for the above, it would be as follows:

$$\text{(possible)} - 10^{1} - 10^{2}\,\Omega\cdot\text{cm}/10^{3} - 10^{4}\,\Omega\cdot\text{cm (not possible)}$$

Later, developmental research was undertaken to add substances that give conductivity to ceramics components, and the developed process was published through technical reports and advertisements. Summaries of such literature and information are shown in Table 5. From the point of view of efficient electrical discharge work, the wider the material's conductive zone, the more desirable the material is, but we also have to consider heat resistance and corrosion resistance.

## 7. Laser Work of Ceramics

Since laser work is a heat process, it is not suitable for glass but is applicable to ceramics. The lasers used to process ceramics are:

(1) Gas laser of $CO_2$ (carbon dioxide)
(2) Solid laser of YAG (yttrium, aluminum, garnet)

The laser is used in ceramics for cutting (including grooving to cut), drilling, and trimming, etc. As these works produce melted wastes, it is necessary to remove the wastes.

Table 5. Ceramics Which Can Be Processed by Electrical Discharge

| Information source | Material composition | Distinction by O and X |
|---|---|---|
| 1. Toyota Institute of Technology | SiC | O (reaction sintering) |
| 2. Nagoya Industrial Research Institute | SiC | O (reaction sintering) |
| 3. Hitachi, Ltd. | SiC | X (HP products) |
| | SiC+TaC, ZrC, WC, $ZrB_2$, $HfB_2$, $HbB_2$, $TaB_2$, TiN | O |
| 4. Electrochemical Industrial Co., Ltd. | BN + $TiB_2$ | O |
| 5. Osaka Industrial Research Institute | $Si_3N_4$ + SiC wisker | O |
| 6. Sumitomo Electric Engineering | $Si_3N_4$ | X |
| | $Si_3N_4$ + TiC, TiN | O |
| 7. Kyushu Industrial Research Institute | $TiB_2$ system | O (tool materials) |
| | C-$B_4C$ | O |
| | C-SiC | O |
| 8. Asahi Glass Co., Ltd. | $ZrB_2$ | O |
| | $Si_3N_4$ + Ti compound | O |
| 9. Hitachi Metals, Ltd. | Sialon + TiN | O |
| 10. Japan Tungsten Co., Ltd. | $ZrO_2$ system + carbide | O |

O mark: electrical discharge machining possible
X mark: electrical discharge machining not possible

There exists compatibility between types of ceramic materials and the laser: for $Al_2O_3$ ceramics the $CO_2$ laser is employed. Recent achievement to punch an $Al_2O_3$ base to form very small holes (by concurrently using NC (computer) controlled X-Y table) is a high-speed technique producing 100 holes/minute.

8. Mirror Finish of Ceramics

Optical glass such as the lens and prism is required to have a mirror surface with an accuracy of up to one-twentieth of the optical wave (0.2 - 0.002 μm for shape and surface roughness, based on the wavelength of visible light as 0.4 μm).

Recently there was a move to make such a lens with pressed glass. If this is to be put into practice, no other material is more desirable than highly heat and corrosion resistant ceramics for the mold. If such a lens is to be made using the mold, we would like to realize it without a grinding process after molding. Kodak (United States) successfully completed this difficult work with its disk camera.

Under the circumstances, it can be said that the needs for mirror finish ceramics have grown sharply.

Nagoya Industrial Research Institute used a conventional grinding wheel (C grit) to make the mirror surface by a diamond grinder and achieved a level of finish which reflects a striped pattern.[11] Also over the past several years Osaka University Faculty of Basic Engineering has been working on honing (the honing work uses a grinding wheel as the grind work does but the work speed is slower than the grinding work) using a diamond grinder to achieve the mirror surface.

It is difficult, however, to apply these two reports to the three-dimensional curved surfaces including the nonspherical surface. The CP Subcommittee members, Saito (Hokkaido University) and Yoshikawa (Tokyo Institute of Technology), and the subcommittee's secretary Kinoshita (Shibaura Institute of Technology, in collaboration with Shibata) have taken up this subject and are working on the theme of development and automation processing technologies for the three-dimensional mirror surfaces including the nonspherical surface. Progress which will lead the world is awaited.[12]

9. Compound Ceramics and Their Processing

Ceramics have major drawbacks because of their brittleness and difficult machinability. There have been many reports published on studies done to improve the brittleness, such as those on $ZrO_2$ ceramics for which partial stabilization is attempted or on SiC wisker addition by Professor Claussen (Humberg Institute of Technology, West Germany).[13]

These improvements in composition, however, cannot give basic shock to the grinding work using a diamond grinder. But considering that glass fiber, carbon fiber, and metal fiber are to be used to make compound materials, we have to be flexible in dealing with the problem.

For raw material pieces coated with ceramics, the above-mentioned processes are in general applicable as the processing method (though it depends on the type of the ceramics used). Also the combination materials, development of which has recently spearheaded by the Japan Metal Society (results are different between ceramics + ceramics, ceramics + metals, compound ceramics + metals), can be treated with the already described processing methods.

For coating and pretreatment (advance processing) of combination materials, members of the Precision Engineering Society have a chance to display their specialty in dealing with electrolysis, mechanochemical work, EEM (elastic emission machining) and also ultrasonic cleaning technology.

10. Examples in Optical Industry

Since I started with glass (grinding) processing and later shifted into the world of ceramics processing, I would like to describe some informative points from the past cases in the optical industry.

Glass as the raw material of the lens and prism has the characteristic of "additivity." This means that the physical/chemical properties and workability of glass can be deduced from its components (contained oxides). Herein lies a clue to improve ceramics workability.

Lately plastic materials were used for optical systems and lenses for spectacles. In general, PMMA (polymethylmethacrylate) used for sunglasses is spray-molded while CR-39 (Columbian resin 39, developed by PPG, United States) used for quality glasses is produced by polymerization in the glass mold. For both cases, the mold is prepared through the NC (computer) control to take into consideration constriction.

In making a mold for ceramics, it is desired to reduce or eliminate, if possible, the cost after baking the ceramic pieces by applying NC technology.

11. Ceramics Composition Design To Improve Workability (Proposal)

In the annual meeting of the Ceramics Industry Association last year (held in Okayama), T. Yamada, director of Niihama Industrial High School, gave a special lecture entitled "Mechanical Characteristics of Fine Ceramics as Seen From Mechanical Design." The point of the presentation was to propose a composition design to control brittleness, a weakpoint of ceramics, and to provide toughness.

In addition to Director Yamada's statement, I would like to propose that workability improvement be included in the composition design.

Ceramics, even with many excellent characteristics when compared to metals and plastics, will come to a halt in expanding application because they continue to have poor workability--a factor which increases cost.

Taking into account this point, Corning (United States) developed machinable ceramics MACOR.[14] In Japan, Mitsui Mining and NGK Spark Plug and several other

companies are following in this field. Property deterioration may be compensated by selective applications but the selling price is still high, posing a problem.

Grinding work technology is advancing as previously stated but grinding is, after all, an expensive processing method. Is it possible to develop new ceramic materials that contribute to lower the cost of the grinding process? I record this as my proposal.

If grinding is said to be the front door (formal approach) to ceramics processing, electrical discharge work would be the back door, and laser work and ultrasonic compound work the side doors. Anyway, I would propose that we learn a lesson from the strategy employed in the attack of Osaka castle and the filling in of the external moat (develop workable composition) before starting the attacks. Therefore, if three parties, the Ceramics Industry Association which is adept in materials, Precision Engineering Society specializing in processing, and Metal Association skilled in joining materials work in collaboration, the basics for ceramics technology in Japan will be established. Then aggressive activities in the application field by such circles as Machine Society and Gus Turbine Society would enable us to fulfill a dream for future fine ceramics binding materials.

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# DEVELOPMENT OF CERAMIC TURBOCHARGERS DISCUSSED

Tokyo CERAMICS in Japanese Oct 86 pp 904-907

[Text] 1. Preface

We will now describe the situation at the time when a method of gas-pressure-sintering silicon nitride was developed in the NIRIM (National Institute for Research in Inorganic Materials) and the process in which NGK Spark Plug Co., Ltd. has succeeded for the first time in the world in developing a ceramic turbo-charger rotor shown in Figure 1. (omitted) by using this method. Therefore, the first half ((Chapter 2.) and the second half (Chapter 3.) have been written by Messrs. Mamoru Mitomo and Yoshinori Hattori, respectively.

## 2. Research on Gas Pressure Sintering Method

### 2.1 What to Research?

In 1972 the NIRIM changed the group research subject from silicon carbide (SiC) to silicon nitride ($Si_3N_4$). Looking back to that time, Hirokichi Tanaka, ex-chief and group leader of the NIRIM at that time, was a man of decision and foresight. But when the group made a start at that time, group members did not know what they should do. This is because it was thought that the silicon carbide research group would conduct research mainly on single crystals and the silicon nitride research group would conduct research mainly on sintering methods. Ceramics which could be obtained at that time were limited to the reaction sintered body and the hot press sintered body. The reaction sintered body with a complex shape can be obtained, but its density is low. On the other hand, the hot press sintered body has high density, but only the hot press sintered body with the simple shape can be obtained. The non-oxide had been called a "Non-Sinterable Substance" because it is difficult to sinter the non-oxide, but the sintering has got popular since GE's Dr. Prochazka succeeded in sintering silicon carbide at atmospheric pressure. If various non-oxide ceramics with complex shapes and high density had been obtained by using the pressureless sintering method, the application of these ceramics to mechanical parts including engines, which had already been proposed in the 1960's, would have been developed.

The "Sintering of Silicon Nitride" was decided as a research theme, because it is considered most important and urgent to develop new materials for ceramics. Research on ceramics was started under conditions in which there was neither sinterable powder nor a sintering furnace. When the research theme was decided, the standard was whether or not this research was a matter worthy of challenge. I started a definite research plan when I was studying sintering work in England in 1973, because I thought that the research was a matter worthy of challenge, even if a half year of work comes to nothing.

2 2 What not to research?

It has long been known that if silicon nitride including a sintering catalyst is hot-pressed, a sintered body will be obtained.[2] Also, there was no wonder when a report on the success of pressureless sintering of silicon nitride was announced, because a report of success on the pressureless sintering of silicon carbide was announced several years ago. Actually, in 1974 the first report on success of pressureless sintering of silicon nitride was announced.[3] Therefore, after I returned from England to Japan in 1974, it was decided not to do research on pressureless sintering, but to study more advanced sintering methods.

Although silicon nitride is a kind of non-oxide, it possesses a feature whereby the relation between thermal decomposition temperature and sintering temperature is closer than that of SiC and AlN. Taking the thermal decomposition temperature, with the dissociated gas-phase of one atmosphere of pressure as $T_0K$, the relation between relative temperature $T/T_0$ and dissociated pressure is as shown in Figure 2. The sintering temperature of each substances is marked with X, and it can be understood that only $Si_3N_4$ has high decomposition pressure, being $10^{-1}$ atmospheric pressures. It can be considered that this high dissociated pressure is probably one of the factors which cause the non-sinterability of silicon nitride. Other substances can be sintered at a relative temperature of about 0.73. To sinter silicon nitride in a condition close to this condition (TN: relative temperature about 0.73), the sintering temperature must be lowered or the thermal decomposition temperature must be raised. To lower the sintering temperature, it is probably necessary to synthesize the sinterable powder and to use a sintering assistant with a low melting point. However, this method was not tested because it was anticipated that the high temperature strength of obtainable ceramics would be low, even if the method succeeded and took a lot of time. Another method is to enhance the thermal decomposition temperature. This method requires heating under high nitrogen pressure from the standpoint of the thermal decomposition reaction shown in the following chemical formula: $Si_3N_4(s) = 3Si(l) + 2N_2(g)$ ----- (1) In this way, a theme is decided, and if what not to do is decided, a direction which should be taken will emerge naturally.

2.3 Development of Gas Pressure Sintering Method

A mixture was formed by adding 5 percent magnesia as a sintering assistant to silicon nitride powder, and a pellet was made from this mixture. As a

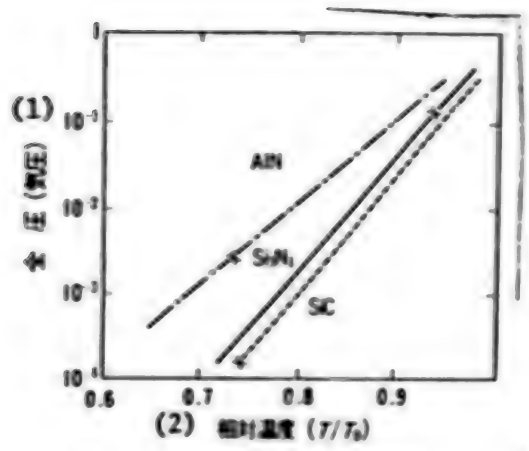


図 2 解離圧と相対温度の関係

Figure 2. Relation Between Dissociated Pressure and Relative Temperature

Key:
1. Total pressure (atmospheric pressure)
2. Relative temperature ($T/T_0$)

result of heating the pellet in nitrogen under 10 atmospheric pressures at a temperature of 1,450 to 1,900°C for 30 minutes, the maximum value was shown at a temperature of 1,800°C, as in Figure 3., and a sintered body with a 95 percent relative density was obtained.[4), 5)] This is a result obtained when no packing coke is used in the sintered body. As a result of heating the pellet in nitrogen under normal pressure, the maximum value was shown at a temperature of about 1,550°C, and the relative density was about 78 percent. As shown in Figure 4, this is because the weight reduction caused by dissociation of silicon nitride is sharp under normal pressure. The weight reduction acts as a growth of pores from the surface of the sintered body, and loweres the density of the sintered body. Objects can be sintered at high temperatures at a high sintering speed, because the dissociation can be controlled in the pressurized nitrogen. This method is called the "Gas Pressure Sintering Method". Although the use of the new method will be more expensive than that of the normal pressure sintering method, it has the merit that ceramics with more excellent characteristics can be obtained.

I can also point out that change and good fortune combined for the development of the gas pressure sintering method.

(1) I was felt relaxed, thinking, "If I fail in this development work, I will be none the worse for it," because I was on the staff of a national research institute, and at that time, not so much was expected from ceramics.

(2) There was no sintering furnace in the national research institute, but there was a furnace used to bring up single crystals in the pressurized gas. Staff members who were conducting research on synthesis of $LaB_6$ single

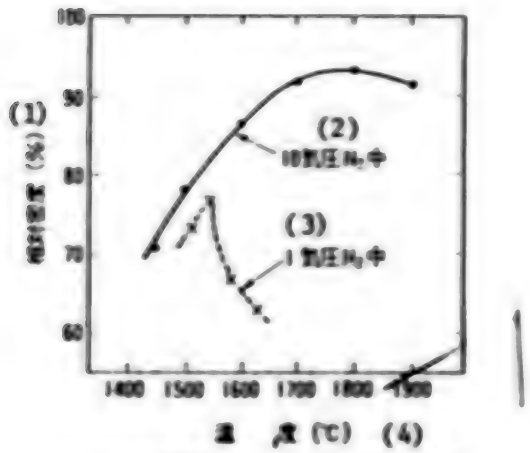


図 3　30 分焼結後の焼結体の密度と焼結温度の関係

Figure 3. Relation Between Density of Body Sintered for 30 Minutes and Sintering Temperature

Key:
1. Relative density (percent)
2. In nitrogen under 10 atmospheric pressures
3. In nitrogen under 1 atmospheric pressure
4. Temperature (degrees centigrade)

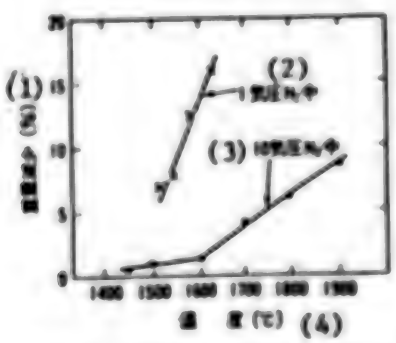


図 4　30 分焼結後の重量減少と焼結温度の関係

Figure 4. Relation Between Reduction in Weight of Body Sintered for 30 Minutes and Sintering Temperature

Key:
1. Reduction in weight (percent)
2. In nitrogen under 1 atmospheric pressure
3. In nitrogen under 10 atmospheric pressures
4. Temperature (degrees centigrade)

crystals willingly approved my use of their equipment, although it was a busy period for their own research.

(3) The sintered body was mixed with 1 or 2 percent silica, and the sinterability was enhanced unintentionally, because I used an agate mortar to mix raw materials, and carried out the mechanical operation over a long period of time.

As shown above, as might be expected by the readers and editors of this journal, there was neither a story of failure nor a story of hard labor in the basic research work. Or rather, it is certain that staff members of the company really were made to suffer unspeakably in putting a sintered body to practical use. (The details of these difficulties can not be made public.)

## 3. Development of Ceramic Turbo-Charger Rotor

### 3.1 Selection of Ceramic Materials

Since the oil shock in the 1970's, world countries have enthusiastically conducted research on high-efficiency engines, and the ceramic material has come into the limelight as a structural material to replace metal. NGK Spark Plug Co., Ltd. has also carried out the development of materials which ought to match future needs. Silicon nitride and silicon carbide were promising as engine ceramics because they are excellent in thermal shock resistance and heat strength. However, there were large projects called, "Improvement of High Temperature Characteristics of Silicon Nitride" and "Increase in Toughness of Silicon Carbide". Just then, it was considered that the gas pressure sintering method developed by the NIRIM would be the best way to correct the defects of silicon nitride. Accordingly, NGK Spark Plug Co., Ltd. received an important technical development subsidy from MITI for this applied research. The research began first with the design of a special-purpose gas pressure sintering furnace, and basic sintering tests were started. These basic sintering tests were conducted using a high pressure furnace made available by the courtesy of Mitsue Koizumi, Professor of the Industry and Science Research Laboratory at Osaka University. After two years of applied research, as initially expected, it was confirmed that the gas pressure sintering method could reduce the amount of sintering assistants, could enhance the density of these sintering assistants, and could produce a sintered body with high reliability. Figure 5. shows the comparison between characteristics of this high pressure sintered body and those of conventional normal pressure sintered body. These characteristics, for engine parts, were judged to be fully suitable, but ceramics had not been used in this field. Work began for the application to auto parts which centered on development of the turbo-charger rotor as the right strategic position for the gas turbine rotor of the future. In addition, in 1984 that NGK Spark Plug Co., Ltd. developed a technology for manufacturing materials for gas pressure sintering silicon nitride on consignment from the RDCJ (Research Development Corporation of Japan), and the company started developing the technology so that it can be put to practical use on a full scale.

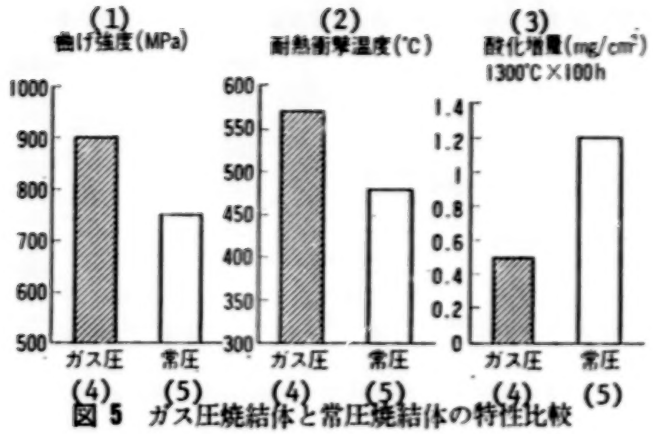


図 5 ガス圧焼結体と常圧焼結体の特性比較

Figure 5. Comparison of Characteristics of Gas Pressure Sintered Body and Normal Pressure Sintered Body

Key:
1. Bending strength (MPa)
2. Thermal shock resistant temperature (degrees centigrade)
3. Amount of increased oxidation (milligram per square centimeter) 1,300 degrees centigrade x 100 hours
4. Gas pressure
5. Normal pressure

3.2 Application to Turbo-Charger Rotor

The turbo-charger is a kind of supercharger, and is designed so that it rotates a turbine rotor by using exhaust gas and can simultaneously supply surplus air to combustion chambers through a compressor rotor mounted on the other end of this turbo-charger, with the aim of enhancing the engine output. The purpose of using ceramic turbo-charger rotors (turbine rotors) is to increase heat resistance and accelerative response (reduction of turbo-lag) by reducing the weight. The turbine rotor of turbo-chargers must withstand the high temperatures and high speed rotation, and must have a metal axle so that a compressor rotor can be mounted on its other end.

The following three important developmental items were established in the development of a ceramic turbo-charger rotor:

(1) Evaluation of technology for manufacturing ceramic rotors and of characteristics of materials, (2) Technology for joining the ceramic turbo-charger rotor with metal, and (3) Evaluation of reliability of the turbo-charger rotor as a rotor.

With regard to a process for manufacturing ceramic rotors, characteristics of basic materials must be used fully in solid rotors. On the other hand, the most important problems were how to make uniform molded bodies in the

molding process and how to make uniform and high-density sintered bodies in the sintering process, because the characteristics of ceramic materials fluctuate greatly depending on molding methods or sintering conditions. An injection molding method was used particularly in products with a complex shape, such as a rotor, because no conventional molding method is suitable. But a large amount of organic binders must be used during molding work, so many problems which were caused by the binders had to be solved. Therefore, the characteristics of a piece cut from a sintered solid body and the material characteristics obtained from a test piece were compared with each other and studied in order to establish the optimum conditions. The process modification carried out by feeding back the results obtained from the comparison and study. The break point of all test pieces was investigated and counter measures were taken, and this work required very much labor and time.

Figure 6. shows the strength distribution of a piece cut from a solid rotor manufactured under the optimum conditions and that of a test piece manufactured by using a general hydrostatic press method. On the other hand, with regard to the joint between ceramics and metal, the study of joint portions was started with consideration to the safety design of cars, because the lubricant of turbo-chargers is used in common with engines. Next, environmental situations under the severest conditions were investigated, and the conditions required in joints were obtained. Conventional methods which have already been developed could not produce resistance to repeated thermal stress, because the metal axle has about four times the thermal coefficient of expansion of silicon nitride. Accordingly, a brazing method was devised which will bring about a stress buffering structure made by combining a number of materials with consideration to Young's modulus, strength, and heat resistance.

Finally, it was necessary to confirm the reliability of the completed rotor. First NGK Spark Plug Co., Ltd. was groping in the dark about how to conduct the non-destructive tests, theoretical evaluation, engine evaluation, etc., and under what methods and conditions. It was very difficult for the company to do these things, because this was the first time for the company to deal with the turbo-charger rotor, and there were no precedents. At the outset, even when the rotor was destroyed during a theoretical excess rotating test, the material, joining and evaluation methods were discussed at all times as to whether they were correct. Accordingly, the causes had to be pursued and the improvements were carried out repeatedly on the basis of the results obtained by investigating the destructive condition, the source of breakage of the destroyed rotor, strength characteristics of materials, the relation between reactivity and strength of joints, and the relation between rotational destruction and operating condition of evaluating units. Therefore, it was necessary to establish all evaluating items and important control items under close cooperation among persons responsible for each process.

Users performed durability tests of the ceramic rotor manufactured in accordance with the above processes, extending over 15,000 hours in total. As a result, the reliability of this ceramic rotor has been confirmed, and it has been demonstrated that the installation of a ceramic rotor on the turbo-charger will raise the acceleration responsiveness by more than 36 percent.

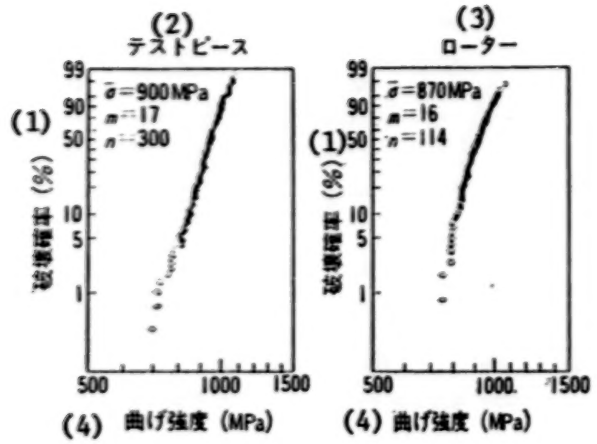


Figure 6. Strength Comparison of Test Piece and Rotor

Key:
1. Rupture probability
2. Test piece
3. Rotor
4. Bending strength (MPa)

The above is an outline of the development of the ceramic turbo-charger. It seems that the success of this development work was favored with such basic technologies as ceramic manufacturing technology, jointing technology, and engine evaluating technology which have long been fostered by NGK Spark Plug Co., Ltd.

Finally, we express our gratitude to persons connected to MITI, RDCJ, and Nissan Motor Co., Ltd. for their cooperation in the development of the ceramic rotor made of gas pressure sintered silicon nitride.

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